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A New Approach to Measuring Pozzolanicity of Supplementary Cementitious Materials Using Existing ASTM Standards

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A New Approach to Measuring Pozzolanicity of Supplementary Cementitious Materials Using Existing ASTM Standards

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Abstract

A New Approach to Measuring Pozzolanicity of Supplementary Cementitious Materials Using Existing ASTM Standards

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Supplementary cementitious materials (SCMs) improve the long-term strength and durability of concrete systems through the pozzolanic and/or hydraulic reactions that form additional calcium silicate hydrate (C-S-H) phases. SCMs come in various shapes and forms, and ASTM C618 provides a standard specification that covers coal fly ash and raw or calcined natural pozzolans. However, the two main criteria outlined by the standard, sum of oxides and strength activity index (SAI), are not sufficient for indicating pozzolanicity of materials; and existing test methods for measuring reactivity or pozzolanicity are yet to be standardized. Due to these existing problems, the accelerated mortar bar test (AMBT) outlined by ASTM C1567 and modified SAI testing were implemented in tandem to assess pozzolanicity of materials. Known inert materials and pozzolanic materials that qualify as Class N pozzolans and Class F fly ash were tested per ASTM C1567 to find replacement levels that suppress ASR expansion below 0.10%. Then the same materials were tested at same replacement levels for modified SAI with a fixed water-to-cementitious materials ratio (w/cm) using cylindrical specimens. The data

from the two test methods were compared and compiled to assess pozzolanicity. Materials that successfully suppressed ASR expansion below 0.10% and passed modified SAI testing over 75% of control were classified as pozzolanic materials. The proposed method of the thesis successfully screened inert materials that qualify as Class N pozzolans, and successfully identified pozzolanic materials.

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Chapter 1: Introduction

Supplementary cementitious materials (SCMs) show an array of benefits when utilized in concrete as partial replacements for ordinary portland cement (OPC). The pozzolanic and/or hydraulic reactions that form additional calcium silicate hydrate (C-S-H) phases have proven to improve the long-term strength and durability of concrete systems, efficiently mitigating possible issues of expansion from alkali-silica reaction (ASR) and sulfate attack (Lothenbach et al., 2011). Pozzolans are defined by the American Concrete Institute (ACI CT-18, 2018) as "a siliceous or silico-aluminous material that will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide at ordinary temperatures to form compounds having cementitious properties (there are both natural and artificial pozzolans)," and the pozzolanic reaction is defined as a "reaction between calcium hydroxide and the oxides in a pozzolan resulting in reaction products having cementitious properties similar to the products that result from the hydration of portland cement." SCMs come in various shapes and forms, but some of the most widely utilized SCMs include Class C and F fly ash, slag cement, silica fume, metakaolin and more. These SCMs are often utilized for their pozzolanicity, with the goal of improving the long-term strength and durability of blended systems. Defined by ACI (ACI CT-18, 2018) as "the finely divided residue that results from the combustion of ground or powdered coal and that is transported by flue gases from the combustion zone to the particle removal system," fly ash is perhaps the most commonly used SCM in concrete systems.

ASTM C618 (ASTM International, 2019c) provides a standard specification that covers coal fly ash and raw or calcined natural pozzolan as Class C, Class F and Class N. The first criterion for these materials is in regard to their chemical composition, where

Class C and F fly ashes need a minimum percentage of 50 in the sum of silicon dioxide, aluminum oxide and iron oxide. Class N pozzolans needs this sum to be at least 70%. Class F fly ashes can have a maximum of 18% calcium oxide, whereas Class C fly ashes need more than 18%. The next criterion is the physical properties of materials, where the strength activity index (SAI) of mortar containing the material under evaluation is tested. The SAI tests compressive strength of mortar cubes with a 20% mass replacement of cement with a SCM at 7 and 28 days, then compares them with control mortar cubes made with 100% cement. For materials to pass the requirements of ASTM C618, either the 7- or 28-day compressive strength must be at least 75% of the control. SAI testing is applied to SCMs to indirectly assess pozzolanicity. The underlying principle is that the blending of SCMs with portland cement improves the strength and decreases porosity of concrete through hydraulic and/or pozzolanic reactions (Lothenbach et al., 2011; Thomas, 2013), compensating for at least some of the strength reduction that occurs due to cement dilution in the mortars. Ultimately, the performance of pozzolans in concrete mixtures, including the effects on compressive strength, is one of the most representative judgment strategy for evaluating supplementary cementitious materials (Pourkhorshidi et al., 2010). Thus, SAI is implemented to assess the extent of pozzolanicity by testing the performance aspect of SCMs by measuring the compressive strength (Donatello et al., 2010; Thorstensen & Fidjestol, 2015).

Studies have shown, however, that the criteria outlined by ASTM C618 – sum of oxides and SAI – are not sufficient for indicating pozzolanicity of materials (Kalina et al., 2019). This is due to the fact that these criteria are not direct measurements of reactivity of materials in concrete or pozzolanicity of materials (Dean et al., 2012; Pourkhorshidi et al., 2010). Sum of oxides does not take into account the distribution of crystalline and amorphous phases of materials, disregarding the mineralogy of the SCMs. Glassy phases are much more soluble than crystalline phases, with quartz being one of the most distinguishable crystalline phases. The SiO_2 in the form of quartz and other insoluble phases cannot be considered as reactive components.

SAI poses even more complications. Not only pozzolanic materials, but inert materials blended with cement could have a significant effect on the hydration of cement phases, the phenomenon that is referred to as a filler effect (Gutteridge & Dalziel, 1990b, 1990a). There are two principal mechanisms that contribute to the filler effect: 1) fillers do not produce hydration products; thus at the same water to solids ratio, the water to clinker ratio is higher, allowing for extra space for hydration products and enabling the growth of more products; and 2) fillers, especially fine materials, provide extra surface area that acts as nucleation sites for hydration products of cement phases (Fernandez Lopez, 2009; Kocaba, 2009; Lothenbach et al., 2011). This poses problems for applying SAI testing in assessing pozzolanicity of materials; since inert fillers and pozzolans both contribute to the formation of additional C-S-H, they both contribute to strength increases making it difficult to differentiate between the two. Furthermore, ASTM C311 (ASTM International, 2018a) specifies the SAI testing procedure using varying water-to-cementitious materials ratio (w/cm) to achieve a flow within 5% of the OPC mortar. Varying w/cm, however, is prone to criticism due to the direct impact it has on compressive strength (Dean et al., 2012; Donatello et al., 2010). The compressive strength of specimens with such materials will depend highly on the w/cm, thus, the use of a constant w/cm in SAI testing has been proposed by studies (Donatello et al., 2010; Pourkhorshidi et al., 2010). Furthermore, there have been suggestions for increasing the strength criteria limit to 80% or even 85% from 75%, based on correlation of cumulative heat testing and compressive strength at 7 days (Dean et al., 2012). Another aspect is the testing at 7 and 28 days, which has been proposed to be increased to 56 and 91 days for high volume fly ash concretes and other sustainable concrete mixtures with lower cement concrete, allowing more time for pozzolanic reactions to occur, possibly preventing premature rejection and false positives (Dean et al., 2012; Kalina et al., 2019).

In spite of its flaws, SAI testing can be implemented successfully to evaluate SCMs with some modifications. First, it must be understood that the test is not a measure of pozzolanicity, but could be a useful predictor of how materials could impact the compressive strength of concrete mixtures. With respect to modifications, using a fixed w/cm of 0.485 could allow for obtaining a more reliable and relevant result. ASTM C109 (ASTM International, 2020b) specifies the w/cm for portland cement mixtures as 0.485, and this could be applied to all mixtures with SCMs. Also, the precision of SAI testing needs improvement due to human-induced errors that occur in the compressive strength testing. As previously mentioned, there have been suggestions to increase the strength criteria limit to 80% or 85%, or to increase the testing dates to 56 and 91 days. However, there is no phenomenological basis for changing the criteria to 80% or 85%, and testing at 56 and 91 days are impractical and take too long, and will not likely be accepted widely.

Because ASTM C618 fails to assess pozzolanicity of materials, a new approach is much needed. There are existing test methods that measure pozzolanicity. The rapid, relevant, and reliable (R^3) method (Avet et al., 2016), calcium hydroxide consumption in pastes using thermogravimetric analysis (Suraneni & Weiss, 2017), the Chapelle test (Snellings & Scrivener, 2016), the Frattini test (Donatello et al., 2010; Snellings & Scrivener, 2016), and a saturated lime test (Donatello et al., 2010) are methods that measure reactivity of pozzolanicity. These are not, however, standardized by ASTM. Determining pozzolanicity of materials for use in concrete as SCMs is crucial, and there is much need for a quick and realistic way to assess materials' pozzolanicity with the standards we have available so that material screening can be quickly implemented.

One proposed way is utilizing ASTM C1567 (ASTM International, 2013), which evaluates the ability of pozzolans to control expansion due to alkali-silica reaction (ASR) reaction within 16 days. ASR is "the reaction between the alkalis (sodium and potassium) in portland cement and certain siliceous rocks or minerals, such as opaline chert, strained quartz, and acidic volcanic glass, present in some aggregates" (ACI CT-18, 2018) and extensive study on the use of SCMs in reducing ASR expansion been carried out (Thomas, 2011; Thomas et al., 2008). SCMs control ASR expansion by reducing the alkalis that are available to the concrete pore solution mainly due to their effect on the composition and alkali-binding capacity of the hydrates (Thomas, 2011). The accelerated mortar bar test (AMBT) standardized in ASTM C1567 can be utilized to determine the efficacy of SCMs in suppressing ASR expansion in concrete. Therefore, it stands to reason that the AMBT should be able to differentiate between pozzolanic SCMs that reduce expansion and inert materials that do not, making it a possible pozzolanicity test. While the general consensus is that the 2-year concrete prism test (CPT) standardized in ASTM C1293 (ASTM International, 2020c) is considered a more reliable predictor of the ability of an SCM to control ASR expansion (Duchesne & Berube, 1994; M. Thomas et al., 2006), the AMBT is a much more convenient method due to its relatively quick results compared to CPT.

SAI and AMBT testing both have shortcomings, yet are widely implemented. The two methods are not direct measurements of pozzolanicity. However, if these existing standards are utilized in a way that they complement each other, this could provide insight into pozzolanicity of materials. Thus, the objective of the thesis is to propose a new approach to measuring pozzolanic activity, utilizing the existing ASTM standard test methods for SAI and AMBT. The remainder of this thesis is as follows. Chapter 2 gives a comprehensive overview on all materials tested and the test methods utilized. Material characterizations were either supplied by the material manufacturer or performed by Dr. Ryan Kalina and Saif Al-Shmaisani. Chapter 3 presents the results from the ASR testing and SAI testing methods outlined in Chapter 2. Chapter 4 then compares the results from ASR testing and SAI testing to classify materials as pozzolanic. Chapter 5 summarizes the research and assesses future challenges, then provides concluding remarks.

Chapter 2: Materials and Methods

To assess pozzolanicity of materials with existing ASTM standard test methods, both inert and pozzolanic materials were tested. The selection of materials was made based on the physical and chemical properties of materials and their classification in accordance with ASTM C618. Classification of materials and characterization testing were compiled from previous research and manufacturer data (Al-Shmaisani, 2017; Al-Shmaisani et al., 2018; Kalina et al., 2019). Section 2.1 details the materials utilized in this study, and Section 2.2 presents the test methods performed throughout this research.

2.1 Materials

The control material for this study was a Type I portland cement in conformance with ASTM C150 specifications from Texas Lehigh Cement Company LP. The same portland cement was used for all testing, herein denoted as OPC. Throughout the research, two types of fine aggregates were used. For SAI testing, graded standard sand in accordance with ASTM C109 and ASTM C778 (ASTM International, 2017) from Ottawa, IL was used as received. For ASR testing, sand from Robstown, TX, determined to be reactive as per ASTM C1260 (ASTM International, 2014b), was re-graded in the laboratory to meet the gradation requirements of ASTM C1567. For both ASR testing and SAI testing, a finely ground quartz powder (Q) was used as an inert filler to compare Class F and Class N materials to a known inert material.

A “production” (fly ashes that do not require additional modifications after recovery from coal-fired power plants that use electrostatic precipitators or mechanical devices such as baghouses) Class F fly ash from San Miguel, TX was used as a known pozzolanic material, herein denoted as F-S. All materials used in this study are shown in Table 2.1 and are designated with the first or first two letters as the material type, followed by a single letter which describes supplier, plant, or material physical characteristics. Two fly ash blends of ASTM C618-conforming Class C and Class F fly ash combined to meet the compositional requirements of ASTM C618 Class F were tested, with one combined by the distributor (BA-V) and another mixed in the laboratory (BA-H). These were tested in order to test Class F fly ashes with CaO contents just under the boundary of Class C fly ashes. A milled bottom ash (MBA) that was ground to a similar particle size distribution as that of production Class F fly ash was tested. There is interest in introducing bottom ash into ASTM C618, but it is known to be less reactive compared to production Class F fly ashes due to a higher degree of crystallinity. Three feldspar and related minerals were tested: dacite (D-S), nepheline syenite (NS-S), and rhyolite (R-O). These materials were determined to be inert in previous research through measurements of calcium hydroxide contents in pastes even though these materials were classified by ASTM C618 as Class N pozzolans (Al-Shmaisani et al., 2018). Five SCMs that showed pozzolanic behavior from a past study (Al-Shmaisani, 2017) were also tested, two of which are reclaimed fly ashes (fly ashes retrieved from disposal sites) and the remaining three remediated fly ashes (fly ashes that are "beneficiated" in order to meet the requirements of ASTM C618 specifications), were also reviewed. Beneficiation is defined (ACI CT-18, 2018) as "improvement of the chemical or physical properties of a raw material or intermediate product by the removal or modification of undesirable components or impurities." All materials chosen for this study encompass a range of materials, some known to be pozzolanic and some not. All of the materials are not traditional sources of Class F and Class N SCMs and would need testing in order to qualify them for use in concrete mixtures, thus making them good candidates for a study to evaluate testing methods for determining pozzolanicity.

Table 2.1: Tested Materials and Designations

Material Designation	Source	Material Classification
OPC	Texas	Type I Portland Cement
Q	West Virginia	Ground Quartz
F-S	Texas	Production Class F Fly Ash
D-S	California	Dacite, Small Particle
NS-S	Arkansas	Nepheline Syenite, Small Particle
R-O	Wisconsin	Rhyolite
BA-H	Texas	Blended Ash, In-House
BA-V	Texas	Blended Ash, VHSC
MBA	Texas	Milled Bottom Ash
RC-G	Texas	Reclaimed Class F Fly Ash
RC-M	Texas	
RM-C	Colorado	Remediated Fly Ash
RM-L	Texas	
RM-S	Oklahoma	

2.1.1 Material Characterization

Data supplied by the manufacturer and obtained by previous researchers were compiled to check materials' chemical and physical requirements according to ASTM C618. Tables 2.2 presents oxide analysis and particle size analysis data, and Table 2.3 presents moisture content and loss on ignition data. Table 2.4 presents results from fineness testing, soundness testing and density; and Table 2.5 presents SAI testing and water requirement of the materials. Materials Q, D-S, NS-S, and R-O all meet the requirements of a Class N pozzolan; and F-S, MBA, reclaimed fly ashes and remediated fly ashes meet the requirements of a Class F fly ash, with the exception of RC-G. However, the only criterion that RC-G fails to meet is moisture content and this can be easily remedied by the supplier through drying. Particle size distribution data for BA-V were not available nor were SAI testing data for BA-H. However, both blended ashes are comprised of production Class C and Class F fly ashes, and meet the oxide composition of Class F fly ash as per ASTM C618.

Table 2.2: Oxide Compositions and Particle Size of Materials of SCMs Used in this Study

Material	**SiO_2 (%)**	**Al_2O_3 (%)**	**Fe_2O_3 (%)**	**CaO (%)**	**MgO (%)**	**SO_3 (%)**	**Na_2O (%)**	**K_2O (%)**	**SiO_2+ Al_2O_3+ Fe_2O_3 (%)**	**d_{10} (µm)**	**d_{50} (µm)**	**d_{90} (µm)**
Q	99.20	0.21	0.13	0.18	0.03	0.09	0.02	0.03	99.54	16.19	16.19	0.32
F-S	62.86	20.26	2.15	5.65	0.58	0.37	2.94	2.21	85.26	5.4	30.1	114.1
D-S	65.47	15.83	5.15	2.80	1.36	0.07	3.70	3.12	86.45	1.6	6.6	40.6
NS-S	57.74	19.48	4.05	1.82	0.99	0.19	5.89	5.85	81.27	2.1	12.9	48.2
R-O	63.26	17.56	4.69	1.69	1.43	0.07	3.60	4.27	85.51	1.6	5.6	38.8
BA-V	45.90	17.88	7.34	16.98	4.02	2.73	1.15	0.97	71.12	-	-	-
BA-H	46.64	20.33	5.05	16.97	4.23	0.94	0.89	0.76	72.02	3.56	20.54	89.10
MBA	59.99	18.43	6.48	9.44	2.15	0.52	0.26	0.91	81.41	4.8	32.8	76.4
RC-G	51.50	21.34	4.92	11.33	2.08	2.72	0.17	0.78	77.77	4.3	22.2	63.5
RC-M	46.95	19.90	8.35	14.05	3.43	0.86	0.77	0.98	75.20	2.6	12.8	59.3
RM-C	59.07	14.07	3.16	9.36	1.57	3.41	3.28	2.94	76.30	2.0	6.3	18.7
RM-L	60.99	13.10	3.08	10.19	0.61	3.92	2.69	3.08	77.17	2.2	9.5	67.0
RM-S	60.13	14.43	4.65	6.91	1.13	3.22	3.20	3.46	79.21	2.3	9.8	60.6

Table 2.3: Results of Moisture Content and Loss on Ignition Testing for SCMs Used in this Study; Red Text Indicates Failure to meet ASTM C618 Criteria

Material	Moisture Content (%)		Loss on Ignition (%)	
Q	0.70	+/- 0.14	0.20	+/- 0.00
F-S	0.65	+/- 0.12	0.36	+/- 0.05
D-S	1.30	+/- 0.10	1.01	+/- 0.10
NS-S	1.32	+/- 0.06	1.57	+/- 0.15
R-O	0.94	+/- 0.06	2.91	+/- 0.02
BA-V	0.74	-	0.67	-
BA-H	0.57	+/- 0.06	0.43	+/- 0.12
MBA	0.73	+/- 0.19	0.29	+/- 0.02
RC-G	3.89	+/- 0.05	0.87	+/- 0.15
RC-M	0.87	+/- 0.03	0.73	+/- 0.07
RM-C	2.87	+/- 0.01	1.79	+/- 0.16
RM-L	1.42	+/- 0.55	1.56	+/- 0.16
RM-S	2.05	+/- 0.06	5.89	+/- 0.1

Table 2.4: Results of Fineness, Soundness and Density Testing on SCMs Used in This Study

Material	Fineness (% Retained)	Soundness (%)	Density (g/cm^3)
Q	1.2	-	2.64
F-S	20.87	0.01	1.78
D-S	6.68	0.00	2.83
NS-S	4.20	0.19	2.71
R-O	7.31	0.01	2.83
BA-V	6.30	-0.06	2.63
BA-H	17.69	0.04	2.24
MBA	18.39	0.01	2.66
RC-G	10.61	-0.02	2.48
RC-M	15.30	0.00	2.69
RM-C	8.70	0.01	2.45
RM-L	15.15	-0.04	2.50
RM-S	10.10	-0.01	2.50

Table 2.5: SAI and Water Requirement for Mortars Prepared with SCMs

Material	**SAI (%)**		**Water Requirement (%)**
	7 Days	**28 Days**	
F-S	76	75	97
D-S	86	81	112
NS-S	75	69	110
R-O	91	93	103
BA-V	93	112	91
BA-H	-	-	-
MBA	87	88	98
RC-G	79	93	100
RM-C	90	113	101
RM-L	89	93	102
RM-S	110	125	102

2.2 METHODS

As discussed in Chapter 1, ASR testing and SAI testing do not directly measure pozzolanicity of materials, but are widely implemented test methods that offer insight into the reactivity of SCMs. The first test method utilized in this thesis is ASTM C1567, the accelerated mortar bar test. The second test method utilized in this study is modified SAI testing with a constant w/cm of 0.485 and cylindrical specimens.

2.2.1 ASTM C1567 Testing

Mortar bars were cast and tested per the ASTM C1567 standard (ASTM International, 2013). Mixture proportions consisted of 1 to 2.25 ratio by weight of cementitious materials to graded reactive fine aggregate. The water to cementitious material ratio (w/cm) was set as 0.47. Initial cement replacement ratio by SCM started at 20% or 25% by mass; and if expansions were near or over 0.10% at 14 days, the replacement ratio was increased. The dimensions of the bars were 25 x 25 x 285 mm (1 x 1 x 11 1/4 in.), with gauge studs placed at both ends with a gauge length of 254 mm (10 in.). Specimens were cast in two equal lifts, compacted with a tamper into the corners as well as around the gauge studs. Cast specimens were then stored in a temperature-controlled room at 23 ℃ (73 ℉) and 100% RH for 24 ± 2 hours. Then the length of the specimens was measured after removing the molds, the readings being set as initial readings. The specimens were fully immersed in tap water and placed in an oven at 80 ℃ (176 ℉) for 24 ± 2 hours, then measured for zero readings. After this procedure, the mortar bars were placed in 1 N NaOH solution that had been preheated in the oven for 24 hours. From the moment of zero readings, specimens were measured at 3, 7, and 14 days, otherwise being fully immersed in the NaOH solution and kept in the oven. At 14 days, specimens with expansion less than 0.1% were deemed to have passed the test, and the process continued for SCMs with increasing replacement ratio until they passed the test.

2.2.2 Modified Strength Activity Index Testing

For modified SAI testing, both standard 50 mm (2 in.) cubic specimens and non-standard 50 mm (2 in.) by 100 mm (4 in.) cylindrical specimens were prepared. The cubic molds were in accordance with ASTM C109 and were prepared by applying a thin coating of form oil to the interior faces of the mold. The mold was then assembled by applying a coating of light cup grease to the surfaces where the halves of the mold join. The mortar mixtures for cubic specimens conformed to ASTM C109. OPC conformed to ASTM C150, and standard graded sand in accordance with ASTM C778 (ASTM International, 2017) was used with a 1 to 2.75 ratio by weight. The w/cm was set at 0.485 for all mixtures. All replacements of OPC by SCMs were by weight, and the replacement amount varied rather than staying at the fixed 20 wt.% dictated by ASTM C311. The mixing procedure was in accordance with ASTM C305 (ASTM International, 2014a) with a mixer also in accordance with ASTM C305. ASTM C109 provides the mixture amounts for 6, 9 and 12 cubic specimens, and the mixture for six cubes was used (500 g of cementitious material, 1375 g of sand, and 242 mL of water) for SAI testing. After mixing, the cubic molds were filled in two equal lifts. Each layer was tamped 32 times in four rounds of eight adjoining tamps at right angles. The open-top surface of the specimens were smoothed using a trowel, then cured in a temperature-controlled room at 23 ℃ (73 ℉) and 100% RH for 24 ± ½ hours prior to demolding. The specimens were stored in the temperature-controlled room at 23 ℃ (73 ℉) and 100% RH until compressive strength testing at 7 and 28 days.

Cylindrical specimens were also prepared for SAI testing because of the poor precision of cubic specimens (Appendix A). The mixing and casting procedures of cylindrical specimens for compression testing were nearly identical as those used for cubic specimens and specified in ASTM C109, except that different molds were used. The plastic cylinder molds with a diameter of 50 mm (2 in.) and height of 100 mm (4 in.) were punctured at the center of the bottom, then taped from the inside to prevent leakage. A thin coat of form oil was applied to the interior surface afterwards. The mixing proportions and procedures conformed to ASTM C109. The standard specifies quantities of materials to be mixed at once for six, nine, and twelve cubic specimens. The volume of a single cube is 125 cm^3 (8 in^3), and the volume of a single cylinder is 196.35 cm^3 (12.57 in^3). Due to the difference in volume between cubes and cylinders, the mixture for nine cubic specimens was used for six cylindrical specimens: 740 g of cementitious materials, 2035 g of sand, and 359 mL of water. For mixing a batch to be used for both cubic and cylindrical specimens, the mixture for twelve cubic specimens was used for 6 cubic specimens and 4 cylindrical specimens: 1060 g of cementitious materials, 2915 g of sand, and 514 mL of water. The mixing procedure conformed to ASTM C305. All of the mixing water was placed in the bowl, and then the cementitious materials were added.

Then it was mixed for 30 seconds at slow-speed. Then over the next 30 seconds, the entire quantity of the sand was added. At the one-minute mark, the mixture was mixed at medium-speed. After that, the mortar was allowed to rest for 90 seconds, then mixing was finished at medium-speed for 60 seconds. Cylinder molds were cast in two equal layers. The diameter of the rod used for rodding was 10 ± 2 mm (3/8 ± 1/16 in), and the mixture was consolidated using 25 strokes per layer. Then a wood float saturated with water was used to finish the top surface. After placing all of the mortar in the molds, the specimens were kept in the temperature-controlled mixing room at 23 ℃ (73 ℉) until the bleed water disappeared. Caps were put on the cylinder, then the specimens were cured in the mixing room for 24 hours. Specimens were demolded and cured in a temperature-controlled room at 23 ℃ (73 ℉) and 100% RH until testing for compressive strength at 7 and 28 days. For compressive strength testing, polychloroprene (neoprene) pads and retainers were used as per ASTM C1231 (ASTM International, 2015b): neoprene pad with a Shore A Durometer Hardness of 50 was used for specimens with compressive strength of 10 to 40 MPa (1500 to 6000 psi), and a neoprene pad with a Shore A Durometer Hardness of 70 was used for specimens with compressive strength to 28 to 50 MPa (4000 to 7000 psi). The compressive strength testing was performed in accordance with ASTM C39 (ASTM International, 2020a). For the purposes of this thesis, results from cylindrical specimens were chosen over cubic specimens due to the higher precision of the results. The comparison of precision for cubes and cylinders, and the decision-making process are presented in Appendix A.

Chapter 3: Results

Chapter 3 presents the results from ASR testing and SAI testing from the methods discussed in Chapter 2. Chapter 3.1 presents the results from testing performed following ASTM C1567. Because the objective of the thesis was to utilize existing ASTM Standard Test Methods to assess pozzolanicity of materials, a wide array of materials that meet the requirements of ASTM C618 as either Class F fly ash or Class N pozzolan were tested following ASTM C1567. The same materials were then tested for modified SAI, and the results are presented in Chapter 3.2. As outlined in Chapter 2, modified SAI testing was carried out with a constant w/cm of 0.485 and cylindrical specimens were used in addition to cubic specimens. Also, instead of testing materials at a 20% replacement of cement by weight, replacement levels that suppress ASR expansion below 0.10% were tested.

3.1 Alkali-Silica Reaction

Control specimens made with OPC and reactive fine aggregate had an average expansion of 0.358 ± 0.005% at 14 days in the AMBT. This data point was used as the expansion value for zero percent replacement level for each of the materials evaluated. Figure 3.1 presents the 14-day expansion of varying substitutions of cement with quartz (Q) by weight. The dashed line denoted as 0.10% is the threshold for specimens to be deemed to have passed the test. Error bars indicate the range of measured expansion results. The error bar for the zero percent replacement level is ± 0.005%, which is smaller than the point marker, thus it is negligible in the plot. The round dotted line represents the least-squares line fit to the data from which the R^2 value is obtained. The plot shows clearly that expansion is suppressed as replacement level of Q increases. From this result, we can first note that a replacement level of 35% Q reduces expansion below the limit specified in the ASTM C1567 method. This suggests that inert materials will reduce ASR expansion if replacement levels are high enough. Figure 3.2 presents the 14-day expansion for F-S. With a traditional Class F fly ash like F-S, expansion is suppressed below 0.10% at both 20% and 25% replacement of OPC by F-S.

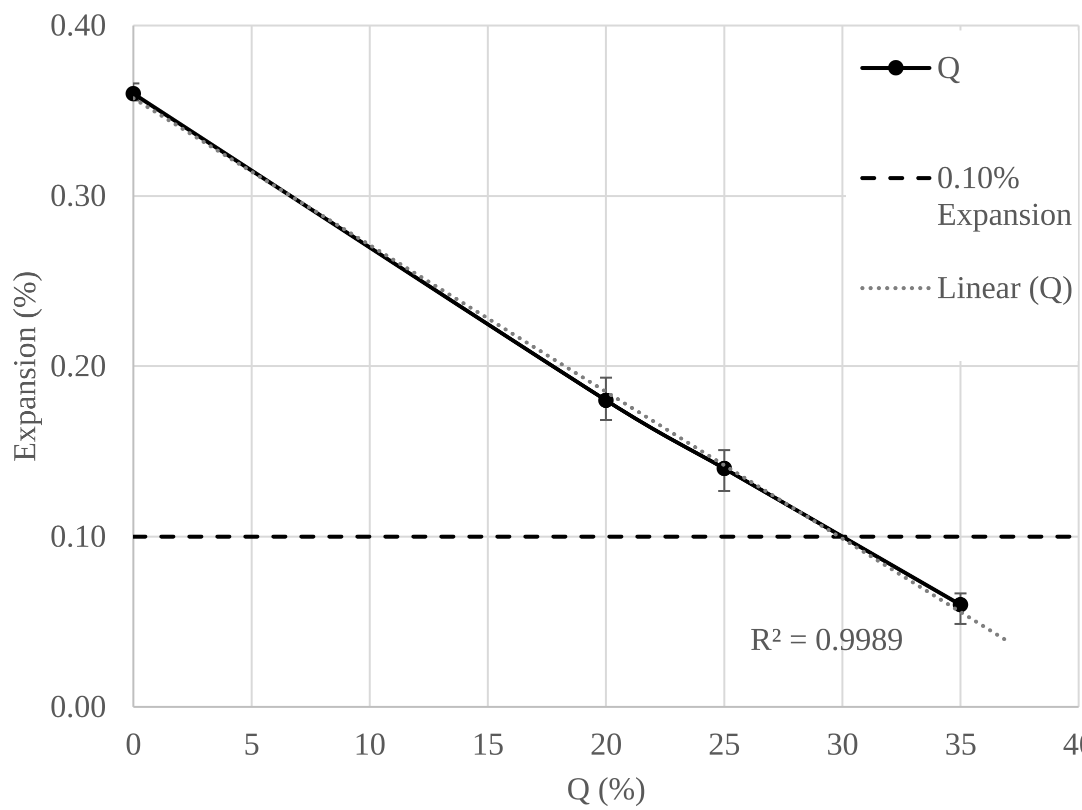

Figure 3.1: ASTM C1567 ASR expansion at 14 days for varying cement replacement levels of Q.

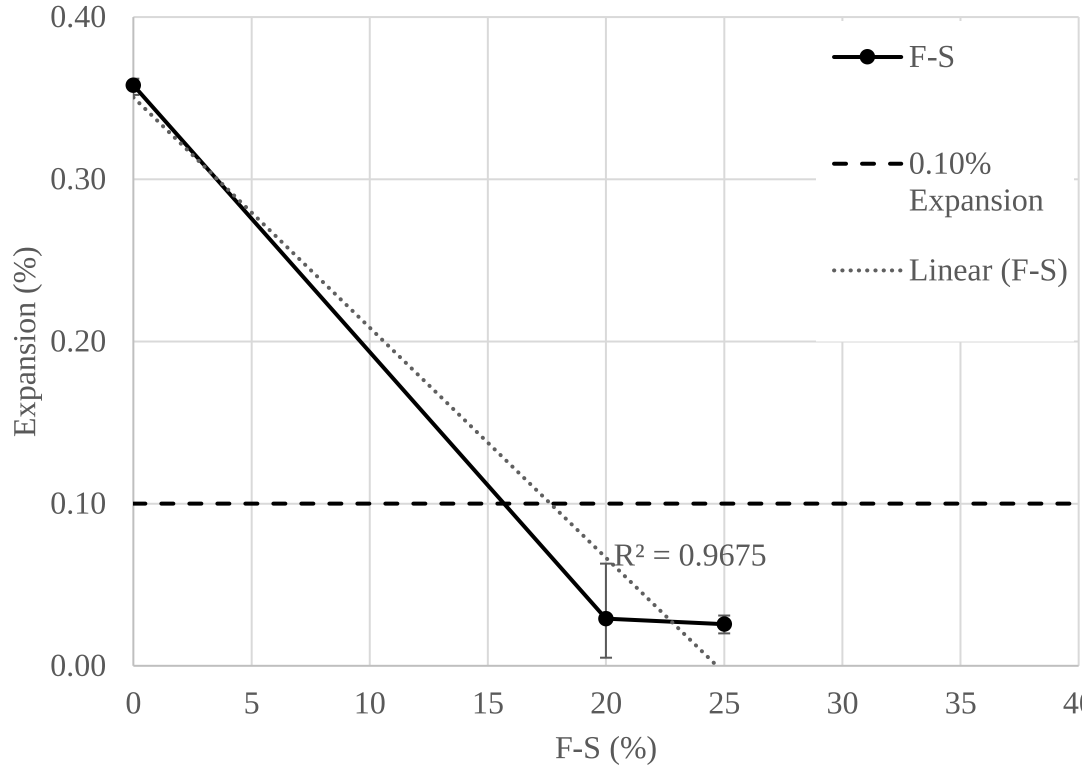

Figure 3.2: ASTM C1567 ASR expansion at 14 days for varying cement replacement levels of F-S.

Figure 3.3 through Figure 3.5 present the 14-day expansion of varying cement substitution levels for D-S, NS-S, and R-O. As stated in Chapter 2, these materials qualify as Class N pozzolans as per ASTM C618, but did not show pozzolanic behavior and are, in fact, inert (Al-Shmaisani et al., 2018). As can be seen from Figure 3.3, similar to Q, D-S is an inert material that passes ASR testing when the OPC replacement amount is high enough. D-S barely passes the ASTM C1567 threshold at 40% replacement with an expansion of 0.090 ± 0.005%. Other inert materials, NS-S and R-O (Figures 3.4 and 3.5), both failed to suppress expansion below 0.1% at 30% replacement, and were projected to also fail at 40% replacement by the least-squares line fit to the data. SCMs are not generally used at replacements greater than 40%, so no further testing was carried out for NS-S and R-O.

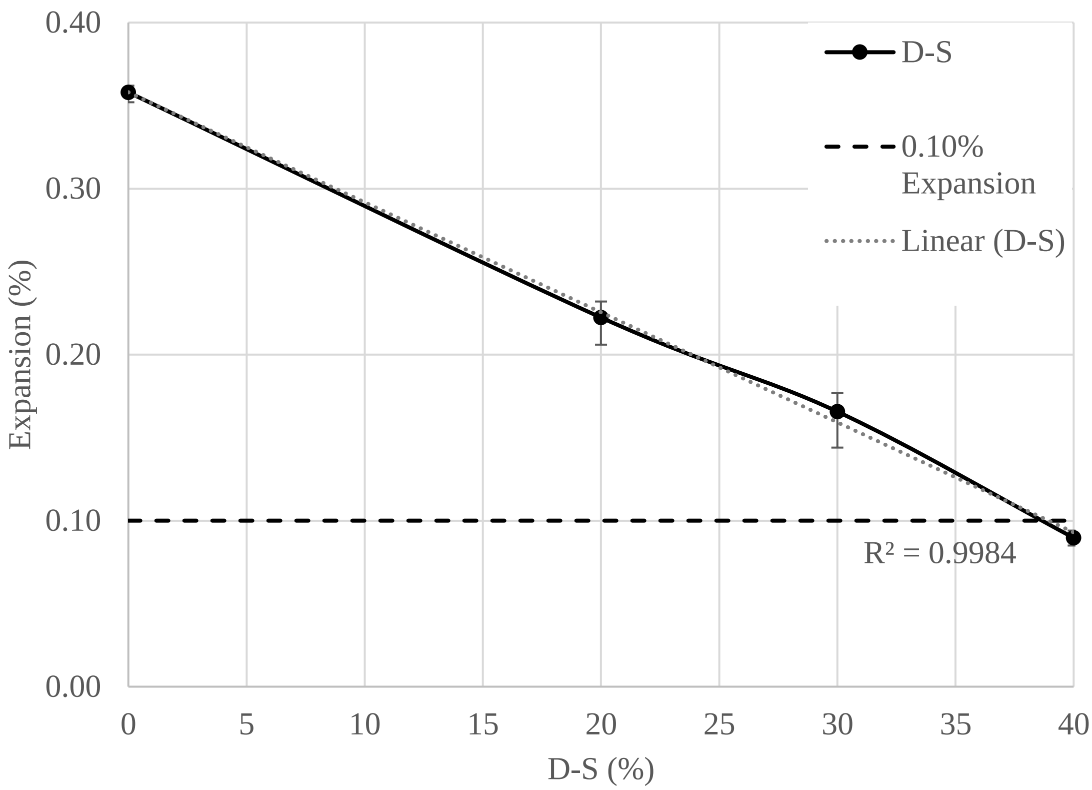

Figure 3.3: ASTM C1567 ASR expansion at 14 days for varying cement replacement levels of D-S. The error bar for 40% replacement level is smaller than the marker.

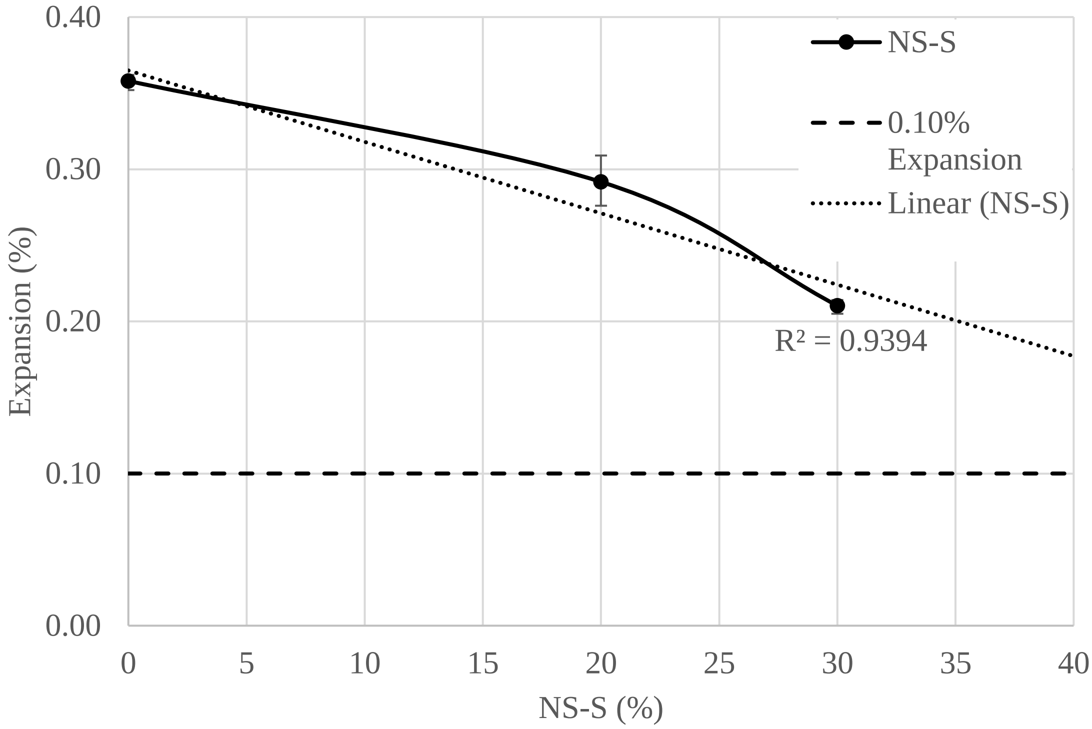

Figure 3.4: ASTM C1567 ASR expansion at 14 days for varying cement replacement levels of NS-S.

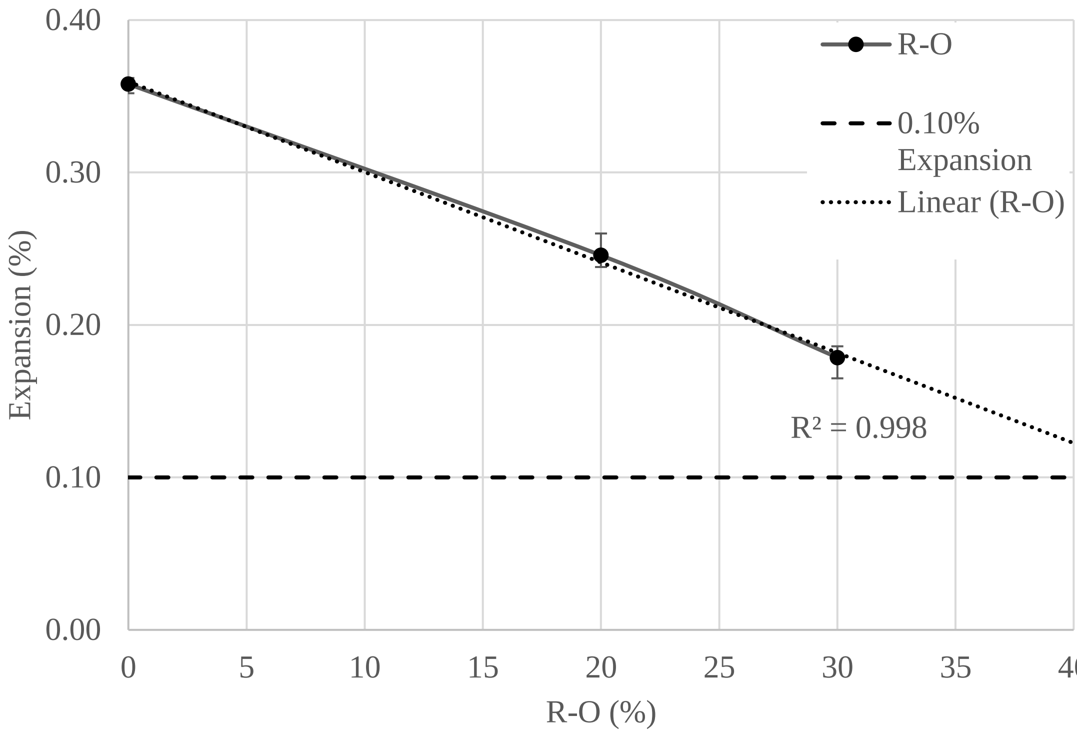

Figure 3.5: ASTM C1567 ASR expansion at 14 days for varying cement replacement levels of R-O.

ASR testing results for BA-V and BA-H are presented in Figures 3.6 and 3.7. As stated in Chapter 2, both blended ashes are a mix of production of Class C and Class F ashes that conform to the sum of oxides for a Class F material as per ASTM C618. With BA-V, the expansion at 14 days with 25% replacement level resulted in an expansion of 0.094 ± 0.004% which was too close to the threshold of 0.10% when taking into account significant figures. A replacement level of 35% suppressed expansion well below the threshold, with an expansion of 0.022 ± 0.001% at 14 days. As presented in Figure 3.7, a replacement level of 25% by BA-H resulted in a 14-day expansion of 0.084 ± 0.006%, safely suppressing expansion within significant figures and error. Figure 3.8 presents the ASR testing result for milled bottom ash (MBA). A 25% replacement of OPC results in a

14-day expansion of 0.060 ± 0.003%, suppressing expansion adequately for the material to pass ASR testing.

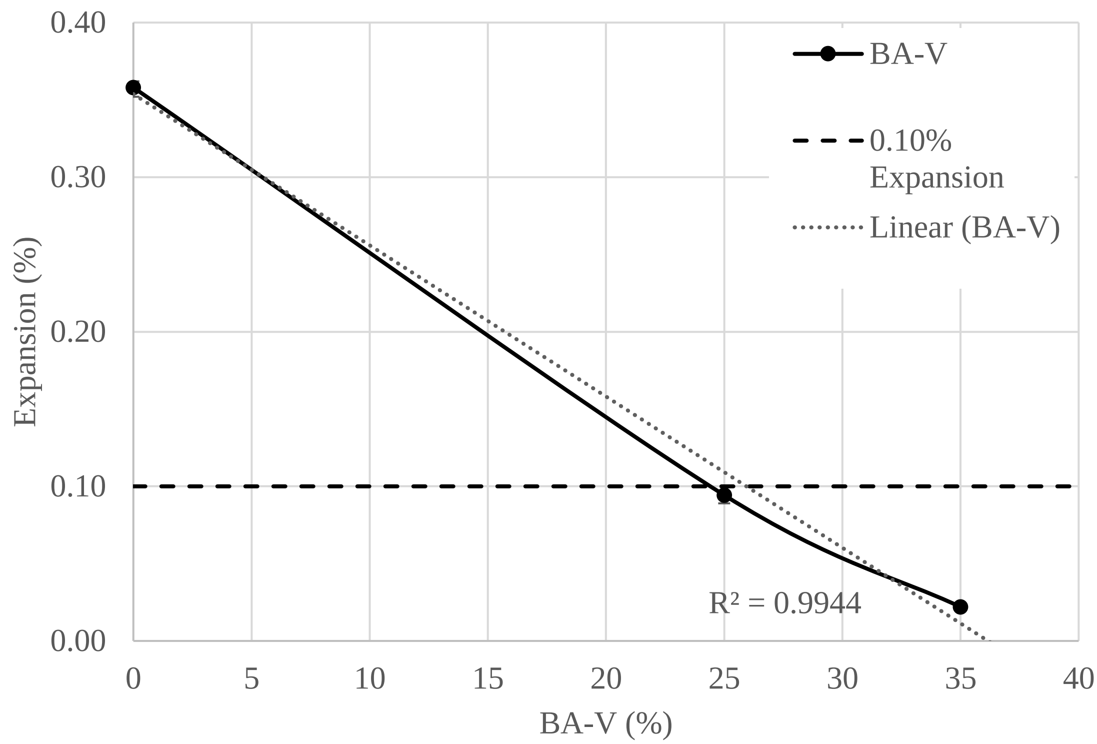

Figure 3.6: ASTM C1567 ASR expansion at 14 days for varying cement replacement levels of BA-V.

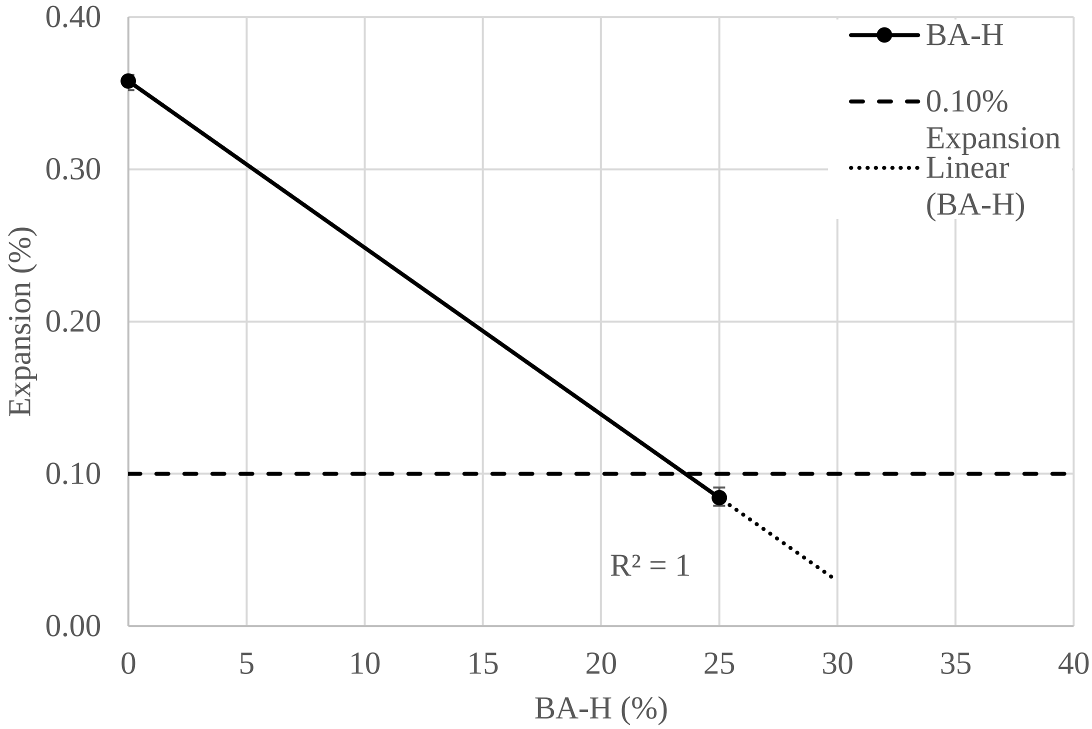

Figure 3.7: ASTM C1567 ASR expansion at 14 days for varying cement replacement levels of BA-H.

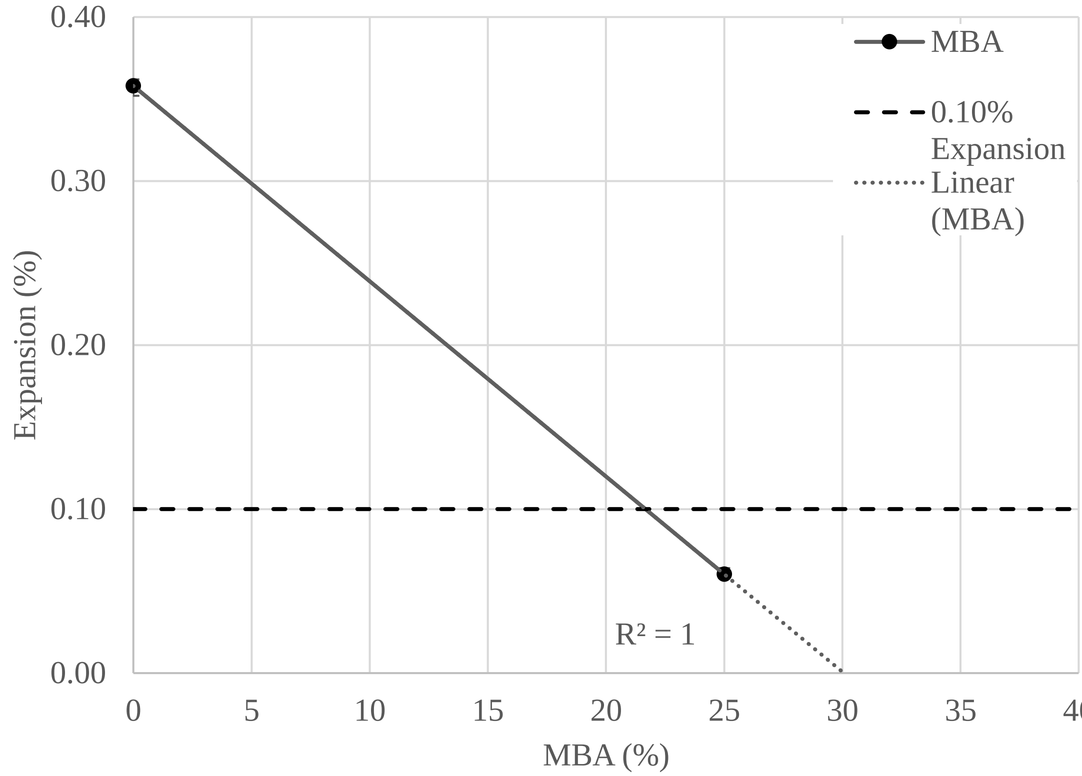

Figure 3.8: ASTM C1567 ASR expansion at 14 days for varying cement replacement levels of MBA.

Figure 3.9 shows the result of ASR testing for reclaimed and remediated fly ashes. These materials were tested at 20% replacement levels, and at 14 days, successfully suppressed expansion below 0.10%. The test results for reclaimed and remediated fly ashes were retrieved from past research, and it was also proven through additional testing that the materials show pozzolanic behavior (Al-Shmaisani, 2017).

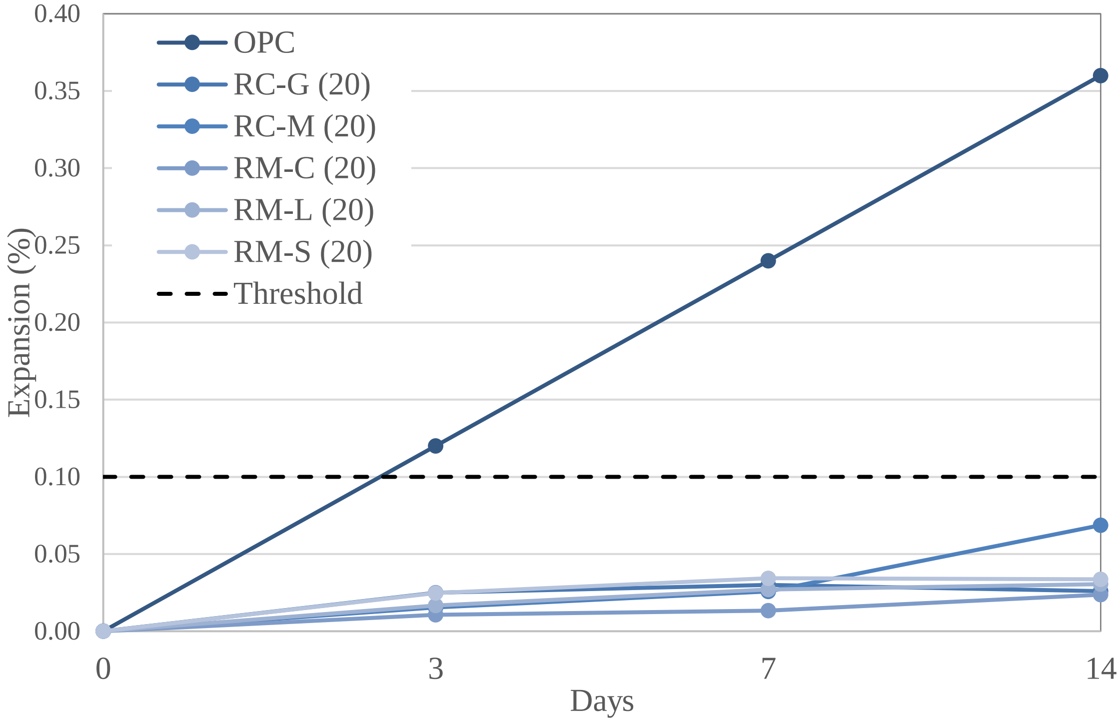

Figure 3.9: ASR expansion of reclaimed and remediated fly ashes at 20% replacement level. Adapted from (Al-Shmaisani, 2017).

3.2 MODIFIED SAI TESTING

Control specimens for SAI testing were made with OPC and graded standard sand and were mixed in three batches (OPC-1, OPC-2 and OPC-3). OPC-1 was mixed for both cubic and cylindrical specimens, thus had two cylindrical specimens for SAI testing for 7-day and 28-day testing; whereas OPC-2 and OPC-3 both had three cylindrical specimens each for 7-day and 28-day testing. The average of all eight cylindrical specimens at 7 and 28 days were 34.1 ± 1.0 MPa (4940 ± 140 psi) and 40.5 ± 0.4 MPa (5880 ± 60 psi), respectively. The range of results at 7 days and 28 days were 2.90% and 0.95% of the average, which are both smaller than the permissible range for two specimens as per ASTM C109. These cylindrical control values were used to evaluate

SAI testing results for all materials. Table 3.1 presents the modified SAI testing results for all materials at select replacement levels tested using cylinders. Text in red indicates that the material failed to meet the minimum 75% strength of control for SAI testing. Please keep in mind that the SAI test is designed for a 20 wt.% replacement of cement with SCMs using variable w/cm, and the data in Table 3.1 represent mixtures with a variety of SCM replacement amounts and fixed w/cm. All materials either "passed" modified SAI testing at both 7 and 28 days, or failed to meet 75% of the control at both 7 and 28 days. The only exception was MBA, which failed to meet the minimum of 75% of control at 7 days, but passed at 28 days. ASTM C618 specifies that a material passes if it meets the minimum at either of the two testing days, thus MBA passes SAI testing.

Table 3.1: Results of Modified SAI testing at 7 and 28 days. Text in Red Indicates Failure to meet Minimum Requirement of SAI Testing

Material	**SAI (%)**	
(% Replacement)	**7 Days**	**28 Days**
Q (30)	70 +/- 1.26	69 +/- 0.33
Q (35)	62 +/- 0.55	64 +/- 0.54
F-S (20)	88 +/- 1.81	100 +/- 1.60
F-S (25)	84 +/- 2.29	96 +/- 0.92
D-S (35)	69 +/- 0.81	70 +/- 2.60
NS-S (35)	65 +/- 0.87	68 +/- 1.08
R-O (35)	65 +/- 0.90	64 +/- 0.46
BA-V (25)	98 +/- 0.61	108 +/- 0.87
BA-V (30)	87 +/- 2.84	101 +/- 0.33
BA-V (35)	83 +/- 1.64	104 +/- 2.03
BA-H (25)	93 +/- 2.42	106 +/- 3.93
BA-H (30)	83 +/- 0.42	101 +/- 1.41
BA-H (35)	82 +/- 0.58	100 +/- 1.52
MBA (25)	71 +/- 1.39*	78 +/- 1.79

* The SAI result does not meet the minimum requirement; however, ASTM C618 specifies that the material passes if the values meet the minimum requirements on at least one of the two testing days.

Table 3.2 presents SAI testing data from manufacturers, and modified SAI testing data from previous researchers. Manufacturer testing was carried out using variable w/cm and 50 mm (2 in.) cubes, shown in Table 3.2 as SAI. The modified SAI testing was carried out with a constant w/cm of 0.485 and were tested as 50 mm (2 in.) cubes. All materials were tested at a replacement level of 20% of OPC by weight, except for BA-H, BA-V, and MBA which were tested at a replacement level of 25% of OPC by weight. The control compressive strength of cubic specimens at 7 days was 35.4 ± 1.2 MPa (5140 ± 170 psi), and 43.0 ± 2.5 MPa (6230 ± 360 psi) at 28 days. Results from the modified

SAI for the reclaimed and remediated fly ashes at a constant w/cm differ from the data supplied from manufacturers that did not use a constant w/cm. The results are generally lower for modified SAI testing compared to the standardized method, which is expected since one can assume that fly ash reduced water demand, enabling a w/cm reduction for SAI testing. The most notable difference from modified SAI testing from cylindrical specimens in Table 3.1 and modified SAI testing from cubic specimens in Table 3.2 was with MBA, where the material passed the lower limit of 75% at 28 days with cylinders but failed with cubes. However, the result with cubes at 7 days had a specimen removed from the averaged data for being outside the permissible range, which could have caused bias in the result (Appendix A).

Table 3.2: Manufacturer supplied SAI data and results of modified SAI testing with cubic specimens for reclaimed and remediated fly ashes. Text in Red Indicates Failure to meet Minimum Requirement of SAI Testing

Material (% Replacement)	**SAI (%)**		**Modified SAI (%)**	
	7 Days	**28 Days**	**7 Days**	**28 Days**
Q (20)	-	-	72 +/- 1.48	81 +/- 1.42
F-S (20)	76	75	82 +/- 1.65	87 +/- 2.37
D-S (20)	86	81	81 +/- 1.63	86 +/- 0.86
NS-S (20)	75	69	73 +/- 0.95	83 +/- 0.86
R-O (20)	91	93	79 +/- 1.17	77 +/- 0.80
BA-V (20)*	93	112	81 +/- 1.61	90 +/- 3.63
BA-H (20)*	-	-	83 +/- 1.80	91 +/- 2.01
MBA (20)*	87	88	57 +/- 1.31	60 +/- 1.99
RC-G (20)	79	93	79 +/- 1.89	92 +/- 1.59
RC-M (20)	-	-	72 +/- 0.02	96 +/- 3.09
RM-C (20)	90	113	92 +/- 3.26	102 +/- 3.01
RM-L (20)	89	93	86 +/- 1.75	80 +/- 2.17
RM-S (20)	110	125	84 +/- 2.89	85 +/- 0.86

* Tested at a replacement level of 25% of OPC by weight in modified SAI testing

Chapter 4: Discussion

Chapter 4 compares the results from Chapter 3 to assess pozzolanicity of materials. ASR testing from Chapter 3.1 demonstrated that ASTM C1567 cannot directly assess pozzolanicity of materials. Increasing the replacement levels of inert materials resulted in suppression of ASR expansion below 0.10%. Because ASR testing and SAI testing cannot directly assess reactivity and pozzolanicity of materials on their own due to their shortcomings, comparing the results from the two methods could provide insight into the suitability of materials as SCMs. The analysis of results from Chapter 3 was done in a two-step process: first, the replacement level that suppresses ASR expansion below 0.1% was determined; then second, the replacement level from the first step was tested for modified SAI testing.

4.1 Comparison of ASR Testing and SAI Testing

From Chapter 3, increasing replacement levels for Q decreased expansion at 14 days, eventually passing the threshold of 0.10% with 35% replacement (Figure 3.1). Figure 4.1 presents modified SAI testing results of Q for varying replacement levels of Q taken from Tables 3.1 and 3.2. Error bars indicate the range of measured compressive strength results to the control. If a material is pozzolanic, the same replacement level for passing ASR testing should also pass SAI testing since the pozzolanic reaction increases strength and reduces ASR expansion. However, modified SAI testing of Q at a 35% replacement level fails to meet the 75% minimum (Figure 4.1). As presented in Chapter 2, Q classifies as a Class N pozzolan by all criteria in ASTM C618 including passing SAI testing at 20% replacement as per ASTM C311 (Table 2.5). However, a 25% replacement level of Q fails to suppress ASR expansion (Figure 3.1), thus a 20% replacement level of Q would also be expected to fail ASTM C1567. This case shows that neither ASR testing nor SAI testing alone can definitively assess pozzolanicity because they can both result in false positives. However, the two methods could complement each other in assessing pozzolanicity by providing checks and balances. For a given material, the following methodology was applied to use ASR testing and modified SAI testing in tandem: ASR testing was first applied to find the specific replacement level of cement for suppressing expansion via ASTM C1567; and with that specific replacement level, modified SAI testing was then carried out to check if the material passes.

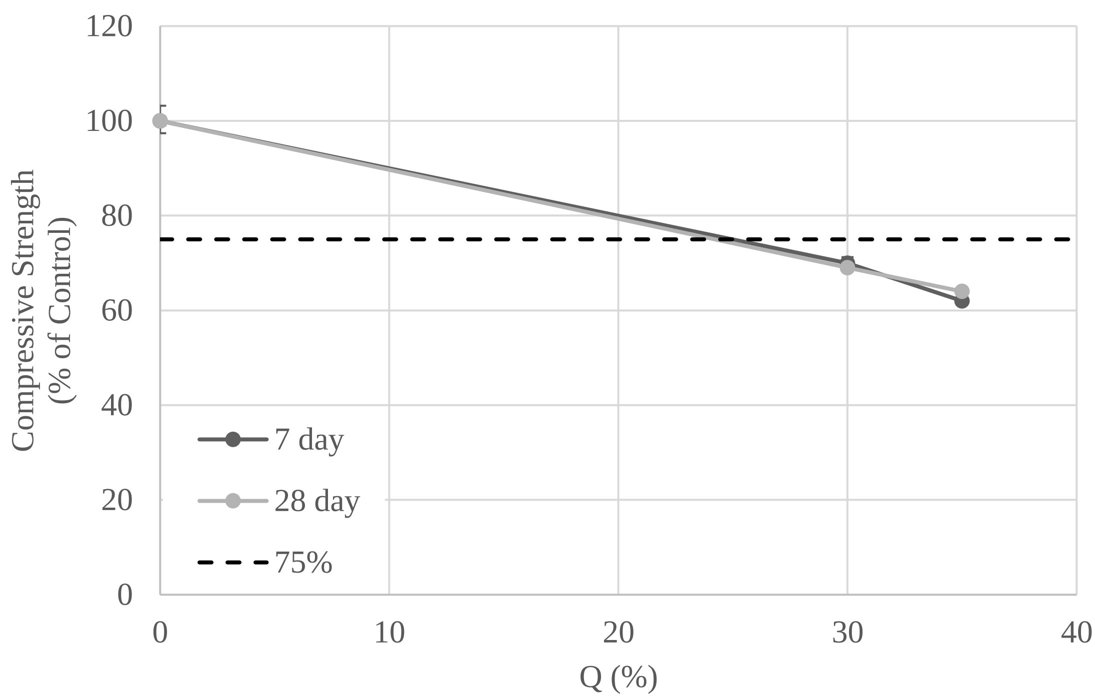

Figure 4.1: Modified SAI at 7 and 28 days for varying replacement levels of Q, with 75% minimum requirement for passing SAI testing.

ASR testing results from Chapter 3 show that F-S, a production Class F fly ash, controls ASR expansion at both 20% and 25% replacement of OPC by weight. The same replacement levels were then implemented for modified SAI testing following our methodology. Figure 4.2 presents modified SAI testing results for F-S, and clearly shows the material passing the minimum 75% of control. Therefore, the combined ASR-SAI method demonstrates that F-S is a pozzolanic material, as expected.

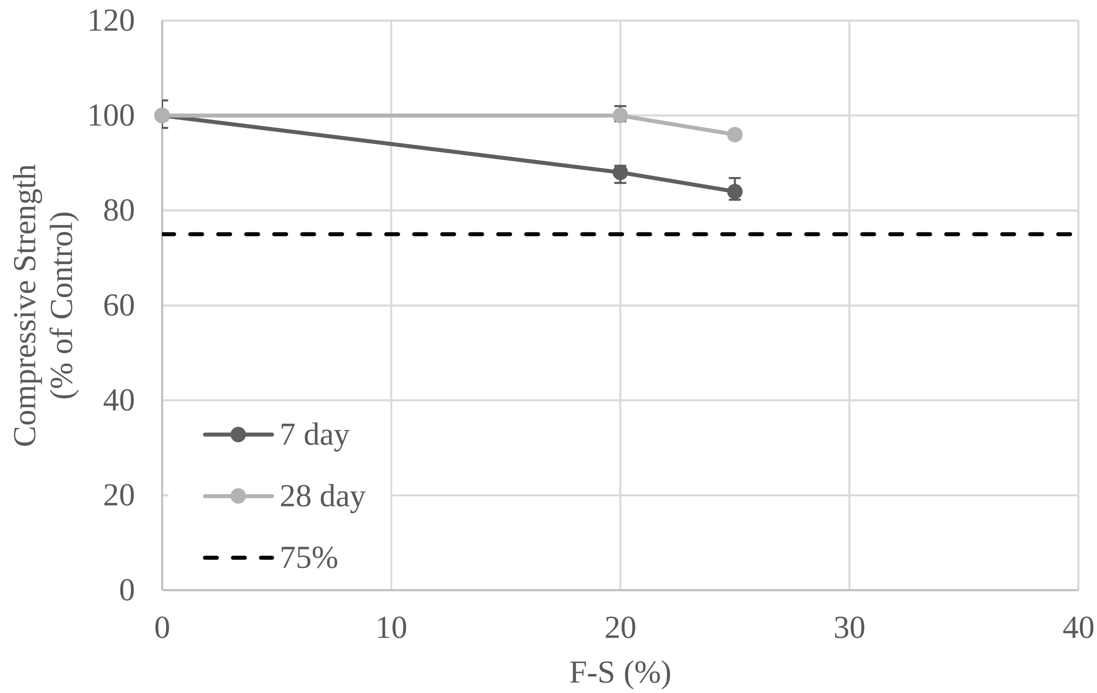

Figure 4.2: Modified SAI at 7 and 28 days for varying replacement levels of F-S, with 75% minimum requirement for passing SAI testing.

From Chapter 3, D-S failed ASR testing at 35% and passed at a 40% replacement level. Figure 4.3 presents modified SAI testing results for D-S at a 35% replacement level showing that D-S failed SAI testing at a 35% replacement level. Applying the same process of first applying ASR testing and then SAI testing, D-S would not be considered pozzolanic. Figures 4.4 and 4.5 present SAI testing of R-O and NS-S. Both materials failed ASR testing at 35% and were projected to fail at 40% by the least-squares line fit to the data. D-S, R-O and NS-S are inert materials that qualify as Class N materials as per ASTM C618 (Al-Shmaisani et al., 2018). Utilizing ASR testing and SAI testing in tandem successfully screens these materials from being considered as pozzolans, avoiding false positives.

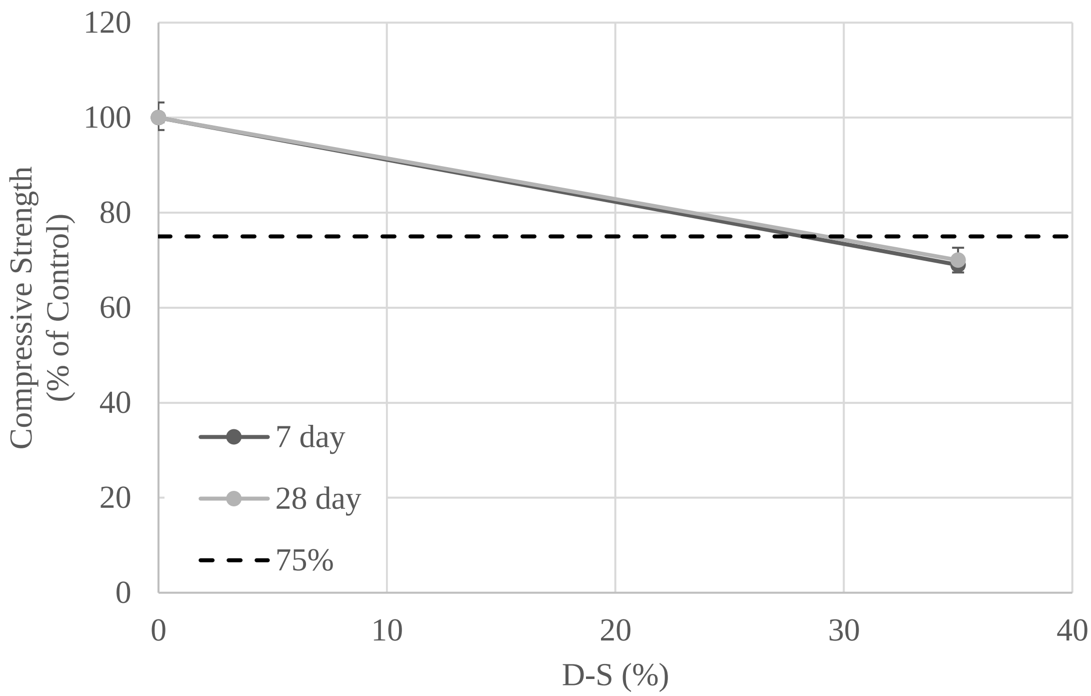

Figure 4.3: Modified SAI at 7 and 28 days for varying replacement levels of D-S, with 75% minimum requirement for passing SAI testing.

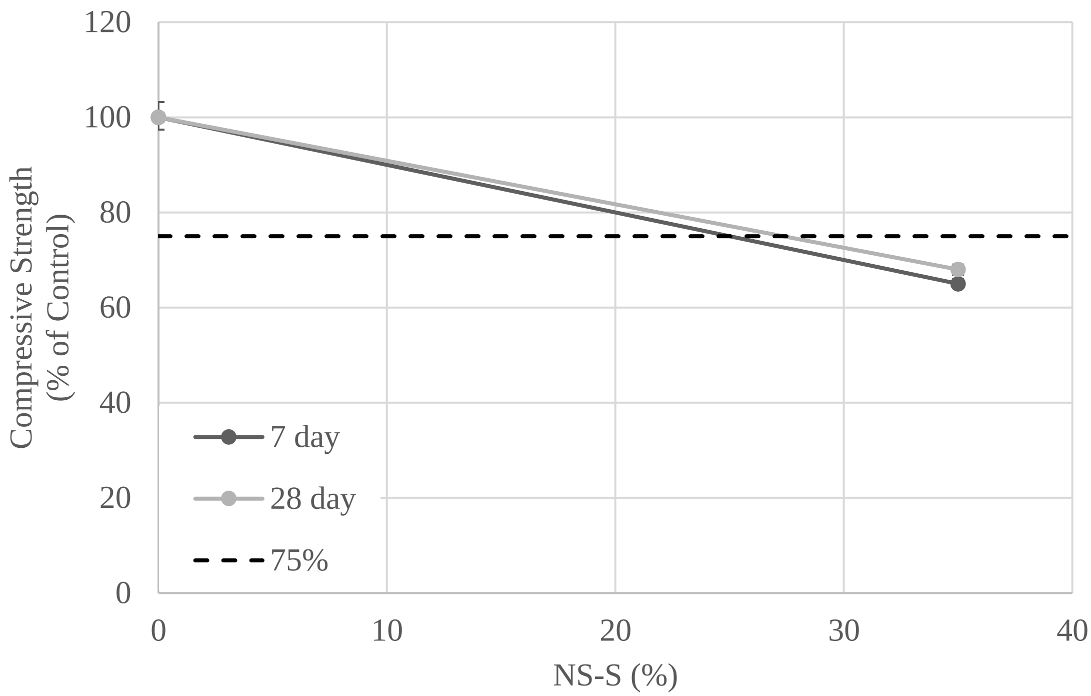

Figure 4.4: Modified SAI at 7 and 28 days for varying replacement levels of NS-S, with 75% minimum requirement for passing SAI testing.

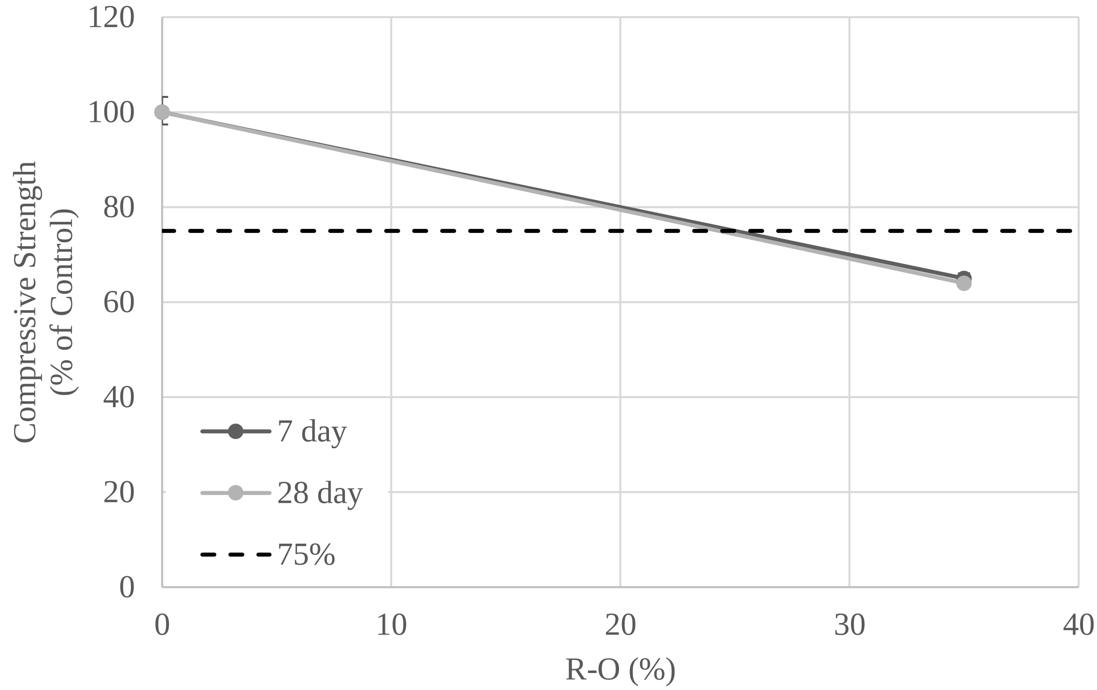

Figure 4.5: Modified SAI at 7 and 28 days for varying replacement levels of R-O, with 75% minimum requirement for passing SAI testing.

Figures 4.6 and 4.7 present modified SAI testing for BA-V and BA-H. BA-V suppresses ASR expansion to the threshold at 25% replacement, and safely does so at a 35% replacement level. BA-H also passes ASR testing at 25% replacement level. Both blended ashes pass SAI at 25, 30 and 35% replacement level. As presented in Chapter 2, both blended ashes are mixes of production Class C and Class F fly ashes that conform to the oxide analysis requirements of Class F fly ashes. By applying the same methodology of utilizing both ASR testing and SAI testing, the blended ashes would be considered pozzolanic.

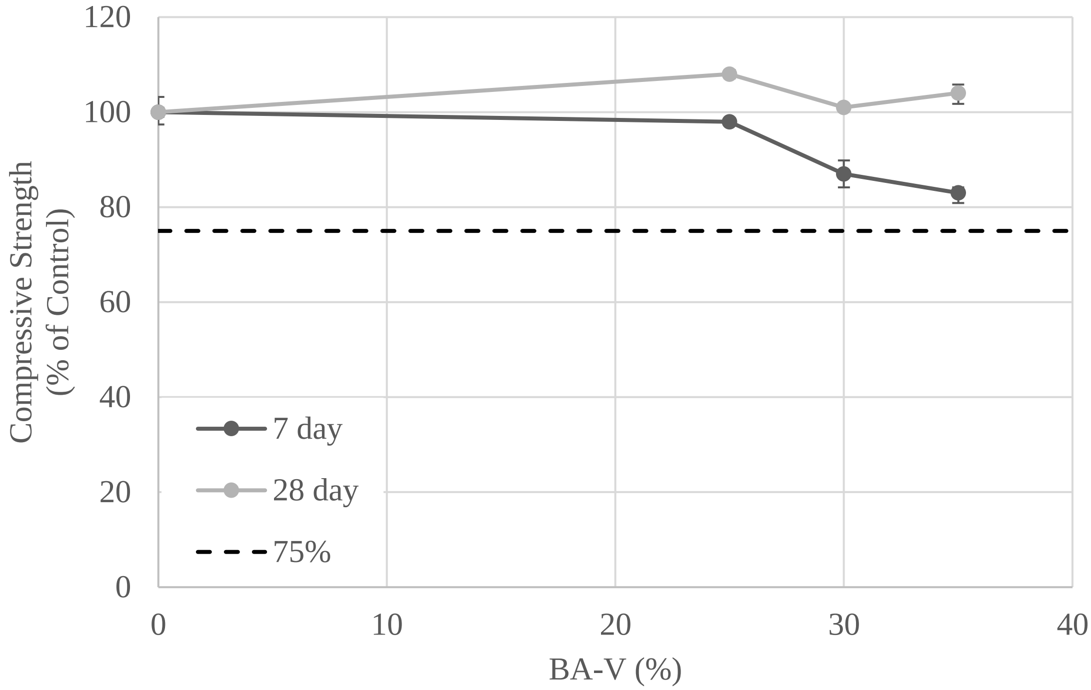

Figure 4.6: Modified SAI at 7 and 28 days for varying replacement levels of BA-V, with 75% minimum requirement for passing SAI testing.

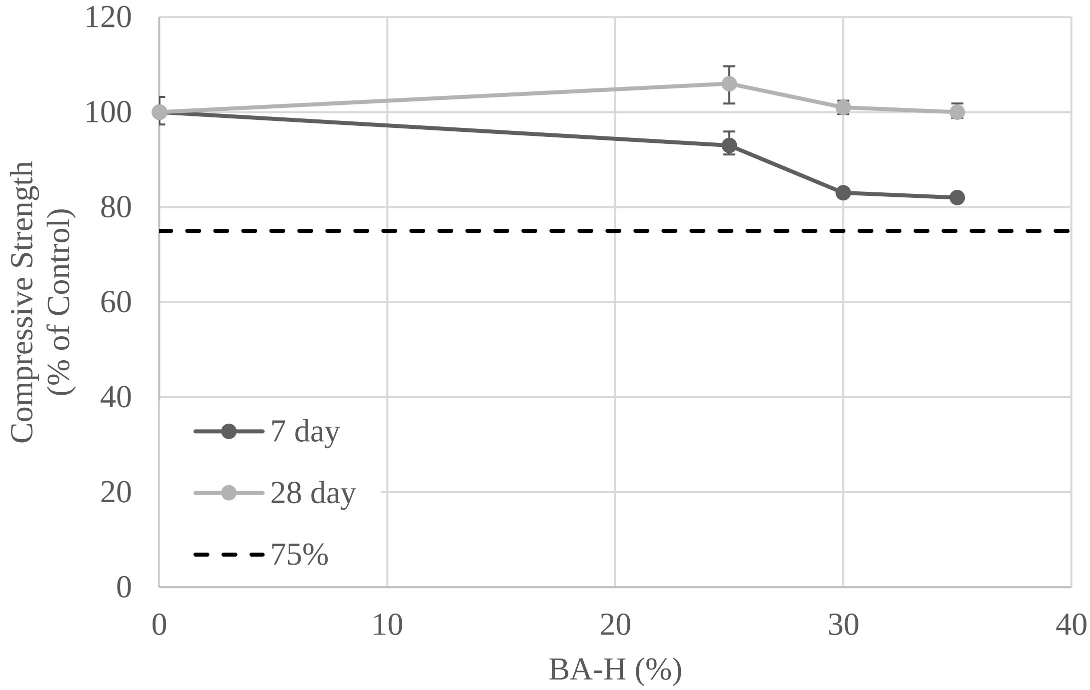

Figure 4.7: Modified SAI at 7 and 28 days for varying replacement levels of BA-H, with 75% minimum requirement for passing SAI testing.

Figure 4.8 presents modified SAI testing of MBA at 25% replacement level. MBA suppresses ASR expansion at a 25% replacement level. While MBA fails to meet the minimum strength of 75% of control at 7 days, MBA passes modified SAI testing with 78 +/- 1.79% of the control at 28 days. Therefore, the combined ASR-SAI method demonstrates that MBA is a pozzolanic material.

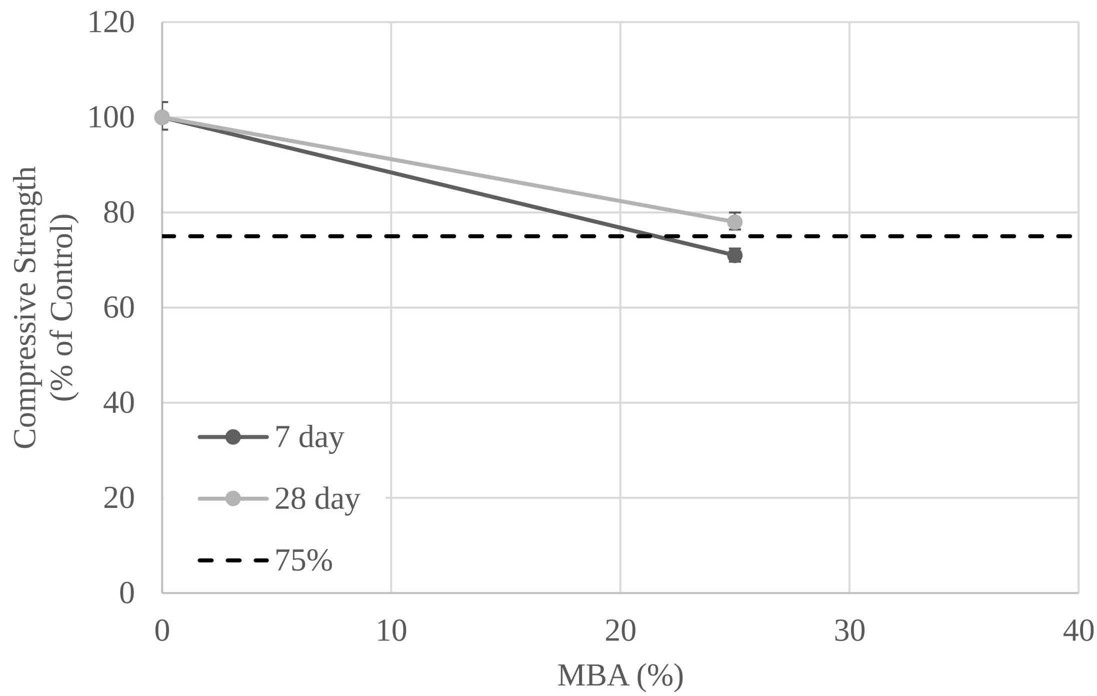

Figure 4.8: Modified SAI at 7 and 28 days for varying replacement levels of MBA, with 75% minimum requirement for passing SAI testing.

Table 4.1 presents results from ASR expansion at 14 days from ASTM C1567 testing and modified SAI testing for reclaimed and remediated fly ashes. As discussed in Chapter 2, the reclaimed and remediated ashes all qualify as Class F fly ash according to ASTM C618, and their pozzolanic behavior was covered in past research (Al-Shmaisani, 2017). As discussed in Chapter 3, these materials successfully passed ASR expansion testing with 20% replacement of cement by suppressing expansion below 0.10% at 14 days. Modified SAI testing at the same replacement levels also resulted in reclaimed and remediated fly ashes passing the criterion 75% of the control strength. Because reclaimed and remediated fly ashes pass both ASR testing and modified SAI testing, the materials can be deemed pozzolanic, which confirms the results of prior research.

Table 4.1: ASTM C1567 ASR expansion at 14 days and modified SAI testing of reclaimed and remediated fly ashes. Text in Red Indicates Failure to meet Minimum Requirement of SAI Testing

Material (% Replacement)	ASR Expansion (%)	Modified SAI (%)	
		7 Days	28 Days
RC-G (20)	0.026 ± 0.031	79 +/- 1.89	92 +/- 1.59
RC-M (20)	0.069 ± 0.002	72 +/- 0.02	96 +/- 3.09
RM-C (20)	0.024 ± 0.007	92 +/- 3.26	102 +/- 3.01
RM-L (20)	0.031 ± 0.000	86 +/- 1.75	80 +/- 2.17
RM-S (20)	0.034 ± 0.002	84 +/- 2.89	85 +/- 0.86

4.2 Compiled Data

Using ASR and modified SAI testing in tandem has successfully screened inert and pozzolanic materials in this study. The data from both tests can be compiled and plotted together to categorize materials as inert or pozzolanic. Figures 4.12 and 4.13 present the compiled data of ASR testing and modified SAI testing at 7 and 28 days, respectively, for all of the materials evaluated in this study. Since ASR testing was performed first for each material, points are only plotted for the percentages where materials passed ASR. The dotted lines represent the thresholds from ASR testing and SAI testing. The minimum limit of 75% for SAI testing and the expansion threshold of 0.10% create Zones I through IV on the plots. Zone I is where the control mix is. ASR testing of the control at 14 days is 0.358 ± 0.005% (fail), and SAI testing is 100%. Zone II is where pozzolanic materials are found, since they pass both SAI and ASR. F-S, BA-V, BA-H, and MBA meet the requirements of ASTM C618 for Class F fly ashes and are also in the Zone II pozzolanic region. Inert materials are found in Zones III and IV, since they fail either ASR or SAI. Q and D-S fall in Zone III. From Chapter 3, increasing the replacement levels of Q and D-S for ASR testing resulted in suppression of ASR expansion below 0.10%. These materials, however, did not pass modified SAI testing at such replacement levels. R-O and NS-S failed to suppress ASR expansion at 35% replacement levels and were projected to also fail at 40%. Testing the materials at similar replacement levels for modified SAI testing results in failure to meet the minimum limit of 75%. R-O and NS-S are plotted using ASR expansion at a 30% replacement level and modified SAI testing at 35% replacement level. As presented in Chapter 3, MBA failed modified SAI testing at 7 days but passed at 28 days, so MBA falls in Zone III in Figure 4.12 but Zone II in Figure 4.13. Figures 4.14 and 4.15 present the compiled data of ASR testing and modified SAI testing with cubes at 7 and 28 days, respectively. The reclaimed and remediated fly ashes successfully suppressed ASR expansion below 0.1% at 20% replacement levels, and also passed modified SAI testing at these replacement levels. These materials appear in Zone II, as expected based on prior testing (Al-Shmaisani, 2017).

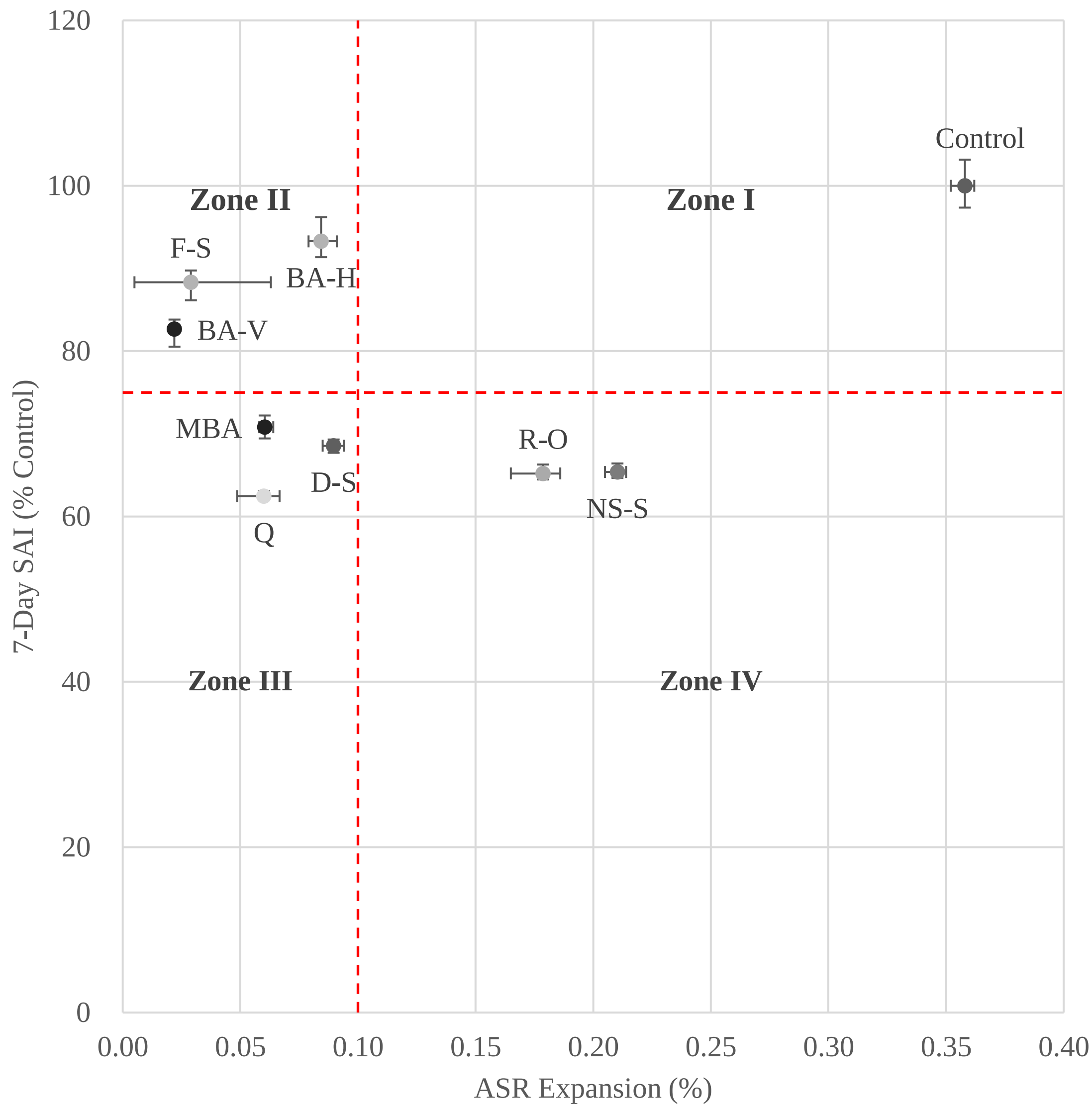

Figure 4.12: Combined data of ASR testing and modified SAI testing with cylinders at 7 days. Zones I through IV are divided by 0.10% expansion from ASR testing and 75% of control compressive strength.

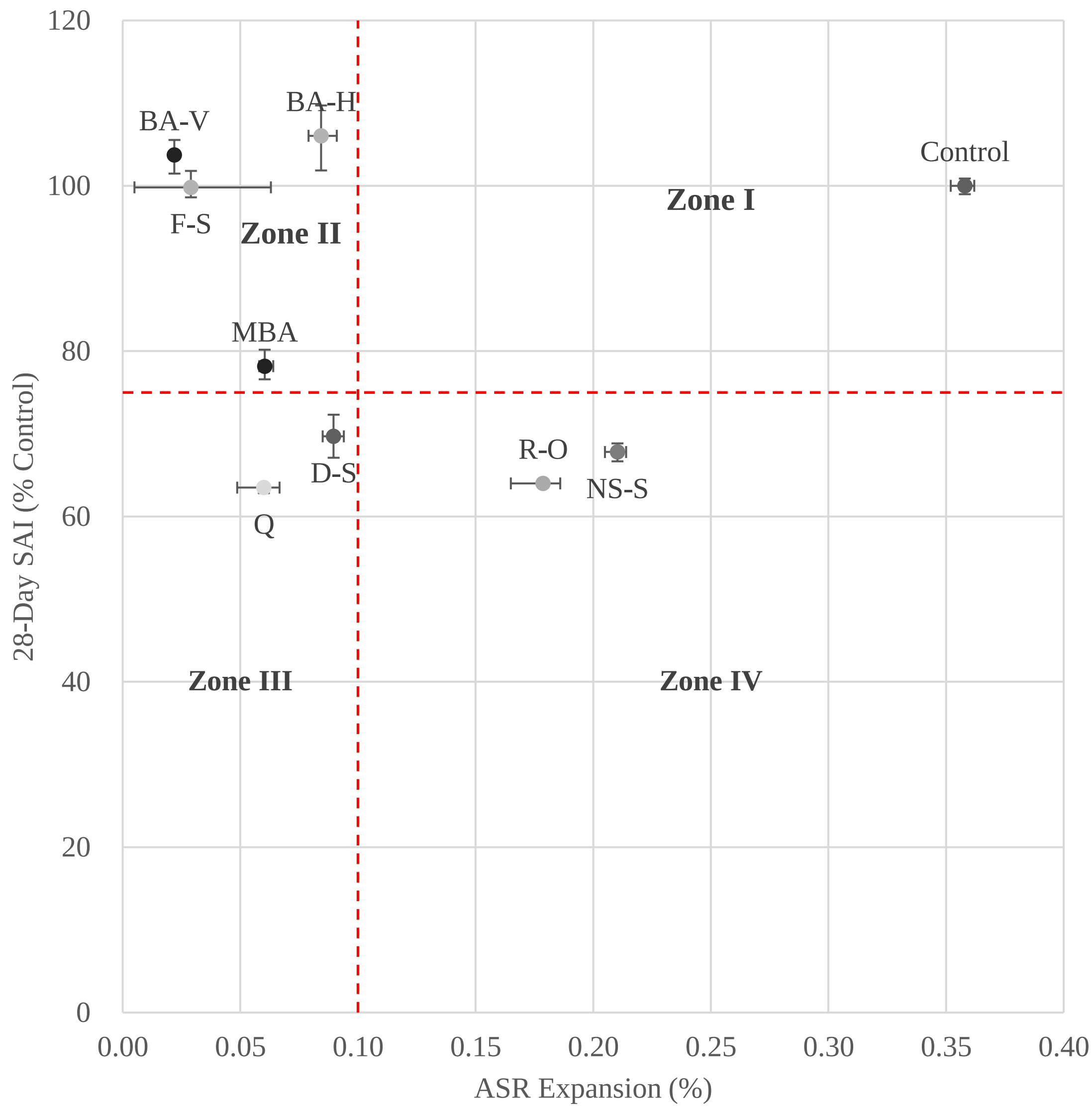

Figure 4.13: Combined data of ASR testing and modified SAI testing with cylinders at 28 days. Zones I through IV are divided by 0.10% expansion from ASR testing and 75% of control compressive strength.

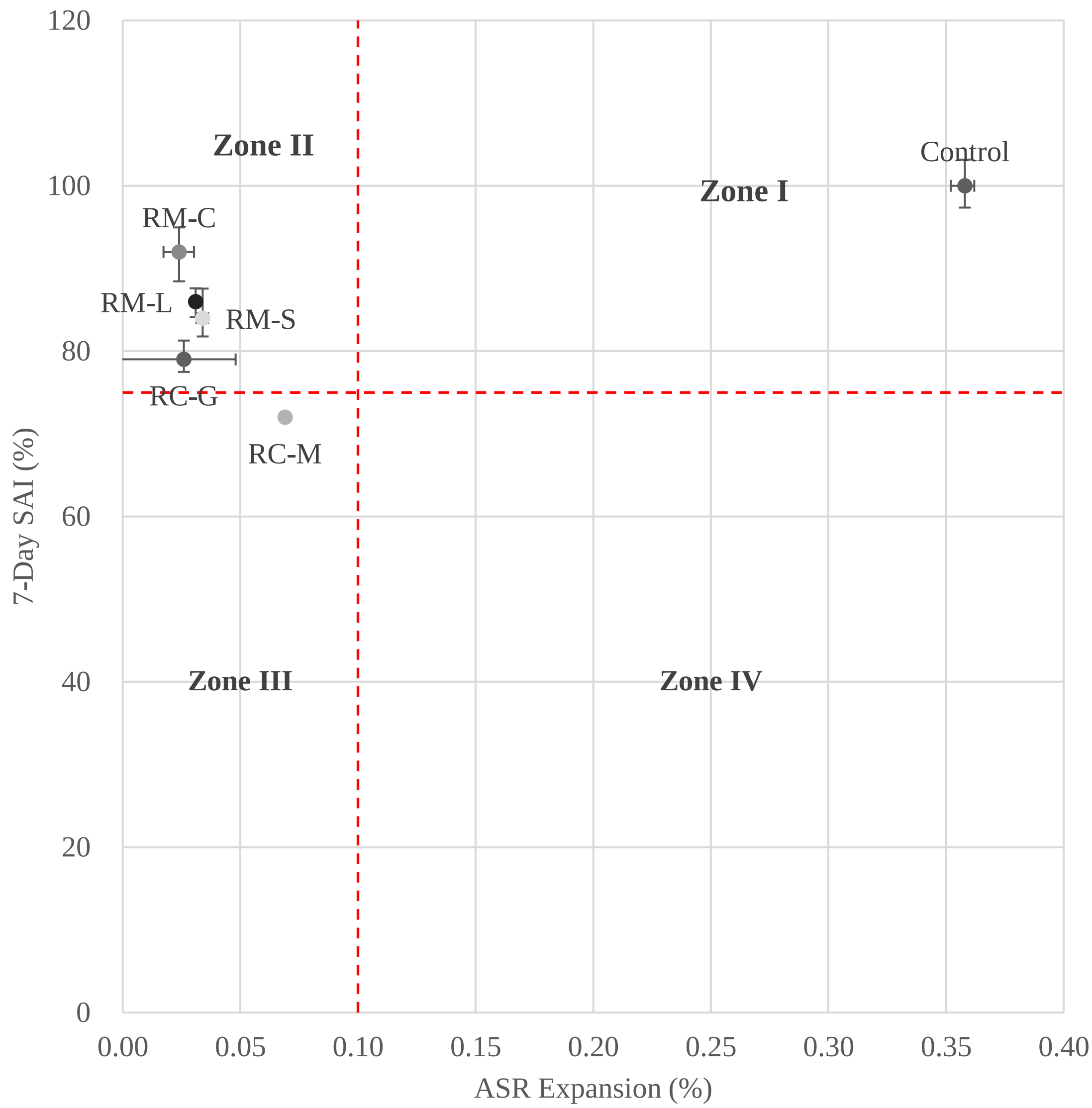

Figure 4.14: Combined data of ASR testing and modified SAI testing with cubes at 7 days. Zones I through IV are divided by 0.10% expansion from ASR testing and 75% of control compressive strength.

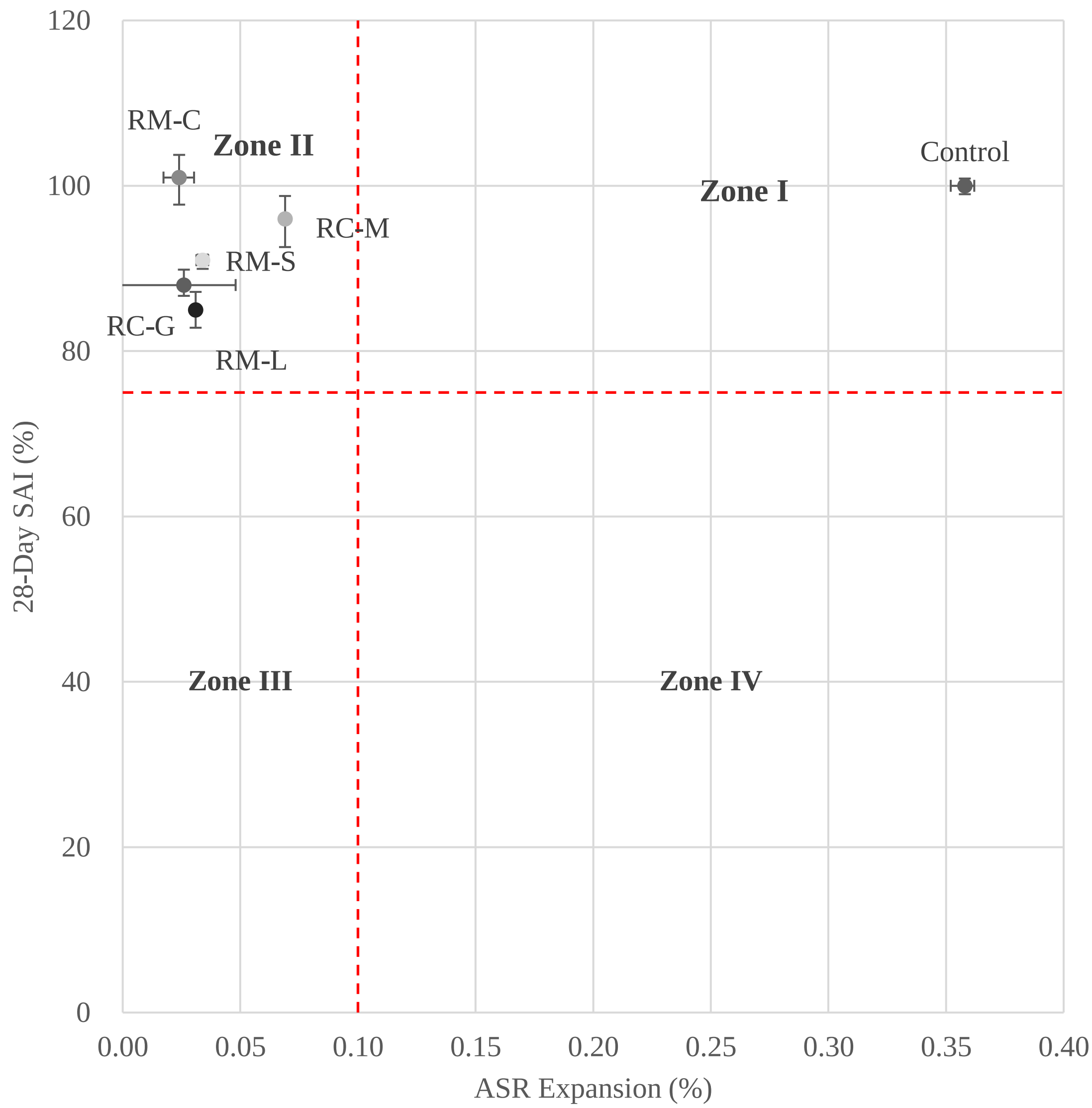

Figure 4.15: Combined data of ASR testing and modified SAI testing with cubes at 28 days. Zones I through IV are divided by 0.10% expansion from ASR testing and 75% of control compressive strength.

Figures 4.12 and 4.13 give insight into how the proposed method of the thesis could be implemented as a quick screening test for materials pozzolanicity. The error bars on the SAI data demonstrate the good precision of using cylinders for testing (Appendix A). Obtaining values with low variability is key for confidence in SAI testing. This is especially true for materials like MBA, where SAI testing results are close to the lower limit of 75%. Furthermore, past research has shown that ASTM C618 alone cannot assess pozzolanicity of materials (Kalina et al., 2019). This is also shown in this research, with inert materials that are classified as Class N pozzolans (Q, D-S, NS-S, and R-O) failing to pass modified SAI at replacement levels that suppress ASR expansion.

The biggest merits of the methodology proposed by this thesis are convenience and accuracy. AMBT testing in accordance with ASTM C1567 takes a total of 16 days to perform, and the modified SAI testing takes 28 days. Implementing the two test methods complements the shortcomings of each method, and provides a way to determine whether or not a certain material is reactive.

Chapter 5: Conclusion

To assess pozzolanicity of SCMs with existing ASTM Standard Test Methods, ASR testing and SAI testing were implemented. ASR testing was done via AMBT in accordance with ASTM C1567; and SAI testing was modified to be tested with a constant w/cm of 0.485 and cylindrical specimens instead of cubic specimens. The first step was to find the replacement level of different SCMs that successfully suppress ASR expansion below 0.10%. Then modified SAI testing was carried out for the replacement levels determined with ASR testing. A material is deemed to be pozzolanic if it passes both tests at a given replacement level. A variety of materials that meet the chemical and physical requirements of Class F fly ash and Class N pozzolans were tested to validate that the method proposed by the thesis successfully assesses pozzolanicity of materials. Chapter 5 provides the conclusion for the research conducted for this thesis. Section 5.1 reassesses the scope of this research and draws conclusions from the test methods; and Section 5.2 discusses recommendations and suggests future work.

5.1 Conclusions

For this study, inert materials that meet the chemical and physical criteria for Class N pozzolans and pozzolanic materials that meet the chemical and physical criteria for Class F fly ashes were tested. The inert materials were finely ground quartz powder (Q), dacite (D-S), rhyolite (R-O) and nepheline syenite (NS-S); and pozzolanic materials were production Class F fly ash (F-S), blended ashes (BA-H and BA-V), milled bottom ash (MBA), reclaimed and remediated fly ashes (RC-G, RC-M, RM-C, RM-L and RM-S).

The proposed method of this thesis successfully screened inert materials. Q passed ASR testing at a 35% replacement level of portland cement by weight, but failed modified SAI testing at the same replacement level. D-S managed to suppress ASR expansion below 0.10% at 40% replacement level, but failed modified SAI testing at a lower replacement level of 35%. Both R-O and NS-S were projected to fail ASR testing at replacement levels higher than 40%, and failed modified SAI testing at 35% replacement level. The four inert materials qualify as Class N pozzolans as per ASTM C618 as discussed in Chapter 2, and the proposed method of the thesis placed the materials in Zone III and Zone IV (Figures 4.12 and 4.13).

The proposed method of this thesis successfully identified pozzolanic materials. F-S suppressed ASR expansion at both 20% and 25% replacement level of portland cement, and also passed modified SAI testing at the same replacement levels. Both blended ashes BA-V and BA-H also passed modified SAI testing at replacement levels that pass ASR testing. MBA passed ASR testing at 25% replacement level and failed modified SAI at 7 days, but passed at 28 days. MBA was successfully identified as pozzolanic by the proposed method and falls in Zone II (Figure 4.13). Reclaimed and remediated fly ashes also fell in Zone II (Figures 4.14 and 4.15), successfully suppressing ASR expansion and passing modified SAI testing at 20% replacement levels.

The new method of determining pozzolanicity of implementing ASR testing and a modified version of SAI testing, based on the conclusions drawn, successfully identified pozzolanic materials and inert materials. The shortcomings of each method are complemented by combining the methods that are prevalent in the field as of now. No single standardized method can successfully determine and measure pozzolanicity of materials, and this new method can offer a viable option until such standardized method is implemented.

5.2 Recommendations and Suggestions

Materials tested for this research varied in their classifications, but the reactivities of these materials were assessed prior to this work. If the proposed method was to be applied to materials that have not yet been determined in their reactivity, further testing needs to be carried out if the actual reactivity is to be determined. ASR testing and modified SAI testing are indirect methods of checking pozzolanicity, not direct methods for measuring pozzolanicity. This work is to be used as a reliable screening method, not a direct measurement of pozzolanicity. In future work, additional testing needs to be performed on the materials screened, including R^3 combined with calcium hydroxide consumption in pastes using thermogravimetric analysis, for example.

For this new approach to determining pozzolanicity of materials to be viable, the accuracy of SAI testing needs to be improved. Using cubic specimens per ASTM C109 is the norm in the cement industry now, but its poor precision and bias potentially can skew results. The implementation of cylindrical specimens has shown benefits in cost, ease of testing, and accuracy. The rather specific limit of 75% compressive strength of control that materials need to meet is an average value, not the range of results from OPC mixes. When there is possibility of bias in the control, results from SAI testing of materials either passing or failing the test could be also biased. The test results from the modified SAI testing with cylindrical specimens provide results with less variability, improving on the existing ASTM C109.

Finding new ways to tackle problems with existing ASTM standards is not an easy process. It can only happen when a problem is prevalent and there is a collective effort to gather data and prove that a change is needed. There is not yet a consensus on the flaws of SAI testing, including the use of a variable w/cm. The usage of cylinders has shown much more precision in SAI testing for this research, but additional testing from other parties would make the case much stronger in order to present an argument for change.

ASTM C1567 accelerated mortar bar method is commonly used, but in predicting the ability of an SCM to suppress ASR, there have been studies suggesting that the method should serve only as a screening test. ASTM C1293 gives a much better indication of pozzolanicity of materials by assessing ASR expansion suppression, but takes a much longer time to perform. The goal was to use existing methods, and if there are ways for ASTM C1567 to be improved, it would help more with the proposed method of this thesis.

Test methods for measuring pozzolanicity are still being developed and trying to find ways into ASTM standards. In theory, a reliable test method that can be widely implemented would be ideal. Until then, the need for screening methods with existing standards are critical, and the testing conducted in this research could very well be such a method.

Appendix A: Cubes versus Cylinders

The shortcomings of SAI testing were covered in Chapter 1. ASTM C311 specifies that specimens be molded in accordance with ASTM C109 (ASTM International, 2020b) and the OPC comply with ASTM C150 (ASTM International, 2019a). By using a fixed w/cm of 0.485, it is possible to obtain a more reliable and relevant result. Another issue that needs to addressed is the actual precision of ASTM C109 itself. The test method is prone to human-induced errors and can result in unreliable data that do not meet the permissible range of specimens from the same batch at the same testing date specified by ASTM C109 (Spencer et al., 2019; Sutter & Bentz, 2017). ASTM C109 specifies that the maximum permissible range between specimens from the same mortar batch at the same test age for three specimens is 8.7% of the average, and for two specimens 7.6% of the average. While it is noted in the standard that the probability of exceeding these ranges is 1 in 100, given that the within-batch coefficient of variation is 2.1%, the result varies depending on the operator. If the range of three specimens exceeds the permissible range of 8.7% of the average, the result that differs most from the average needs to be discarded. Then the remaining two results must meet the permissible range of 7.6% of the average to be compliant to the standard. This, however, means that the true average of three specimens is discarded; and this could create a bias, either on the lower or higher side of the initial average of three specimens.

Due to these shortcomings of SAI testing, modifications are much needed. To assess this problem, SAI testing was modified to have a constant w/cm for all mixes, and cylindrical specimens have been implemented in lieu of cubic specimens. Plastic cylindrical molds are cost-effective, readily available and versatile compared to cubic molds (Pigeon, 2015), and are widely used for compressive strength testing of chemical-resistant mortars, grouts and polymer concretes as per ASTM C579 (ASTM International, 2018b). Cylinder specimens are mainly used for concrete testing as per ASTM C192 (ASTM International, 2019b), and cylinder molds must meet ASTM C470 (ASTM International, 2015a). A cylindrical mold with a diameter of 50 mm (2 in.) and height of 100 mm (4 in.) is included in ASTM C470, but not specified for use in concrete testing following ASTM C192. However, ASTM C579 specifies a test method (Test Method C) for cylindrical specimens with a diameter of 50 mm (2 in.). To assess the issue of precision of SAI testing, 50 mm (2 in.) cube molds and 50 mm (2 in.) by 100 mm (4 in.) cylinder molds were tested to compare the precision of the two methods discussed in Section 2.2.2.

A total of 17 mixtures were tested for direct comparison for cubic specimens and cylindrical specimens. Tables A.1 and A.2 present the compressive strength testing results for cubic specimens and cylindrical specimens respectively. The range of the compressive strength results in percent of the average are also presented. As per ASTM C109, the maximum permissible range between specimens from the same mortar batch at the same test age for three specimens was 8.7% of the average, and for two specimens 7.6% of the average. Specimens that did not meet these criteria are highlighted in red and blue, red for specimens rejected for being too low and blue for specimens rejected for being too high. Three control mixtures (OPC-1, OPC-2 and OPC-3) and 14 mixtures with varying SCMs at different replacement levels were tested. The mixture designations follow Table 2.1, and replacement levels are specified in parentheses. OPC-1, BA-H (30) and BA-V (30) were batched for 12 cubic specimens. The mixtures for 12 cubic specimens were cast into 6 cubic specimens and 4 cylindrical specimens. Other mixtures were mixed in two batches, one for six cubic specimens and the other for 6 cylindrical specimens.

Table A.1: Compressive Strength Testing Results from Cubic Specimens

Material (% Replacement)	7-day #1 (psi)	7-day #2 (psi)	7-day #3 (psi)	7-day Range (%)	28-day #1 (psi)	28-day #2 (psi)	28-day #3 (psi)	28-day Range (%)
OPC-1	4683	5085	4805	8.29	5123	**4675**	5008	8.84
OPC-2	4868	**5428**	5133	11.20	6268	6013	6240	4.13
OPC-3	5313	5240	**4633**	12.89	6228	6135	6478	5.45
F-S (20)	**4610**	4143	4313	11.06	5220	5445	5515	5.47
F-S (25)	**4138**	3698	3618	14.22	5355	5653	5633	5.36
MBA (25)	2858	**2655**	2993	11.54	3783	3858	3610	6.60
Q (30)	3065	**3478**	3158	11.59	4158	4065	4265	4.80
Q (35)	3003	2973	2958	1.55	3815	3720	3785	2.52
R-O (35)	3393	3278	3315	3.46	3938	4148	3830	7.99
NS-S (35)	2825	2758	2910	5.39	3640	3520	3465	4.94
D-S (35)	3208	3223	3225	0.54	**3295**	3948	3895	16.64
BA-H (30)	**4725**	4280	4135	14.02	**6703**	6043	6325	10.67
BA-V (30)	4868	**4545**	5090	10.95	6523	6438	6465	1.31
BA-H (25)	4178	4235	4363	4.34	**6473**	5820	5570	15.85
BA-H (35)	3793	3623	3813	5.08	5455	5933	5638	8.41
BA-V (25)	4260	4095	**4540**	10.65	5328	5780	5738	8.06
BA-V (35)	4460	4615	**4188**	9.42	6768	6370	6423	6.10

Text in red indicates that the specimen was rejected for being too low.
Text in blue indicates that the specimen was rejected for being too high.

Table A.2: Compressive Strength Testing Results from Cylindrical Specimens

Material (% Replacement)	7-day #1 (psi)	7-day #2 (psi)	7-day #3 (psi)	7-day Range (%)	28-day #1 (psi)	28-day #2 (psi)	28-day #3 (psi)	28-day Range (%)
OPC-1	4943	4949	-	0.13	5863	5857	-	0.11
OPC-2	5010	4847	5096	4.98	5889	5908	5815	1.57
OPC-3	4812	4809	5048	4.88	5889	5927	5854	1.24
F-S (20)	4401	4255	4433	4.09	5818	5981	5793	3.20
F-S (25)	4271	4045	4076	5.47	5634	5576	5685	1.92
MBA (25)	3430	3494	3567	3.92	4500	4570	4710	4.58
Q (30)	3452	3513	3389	3.60	4022	4041	4061	0.95
Q (35)	3061	3080	3115	1.75	3755	3691	3745	1.71
R-O (35)	3274	3185	3204	2.77	3732	3787	3761	1.44
NS-S (35)	3280	3194	3213	2.66	3917	3987	4045	3.20
D-S (35)	3344	3424	3392	2.35	4092	4248	3943	7.47
BA-H (30)	4121	4080	-	1.01	6013	5847	-	2.79
BA-V (30)	4420	4140	-	6.55	5917	5955	-	0.64
BA-H (25)	4752	4513	4557	5.18	6446	6261	5984	7.41
BA-H (35)	4073	4041	4099	1.41	5812	5777	5955	3.05
BA-V (25)	4818	4828	4879	1.25	6357	6404	6303	1.60
BA-V (35)	3978	4140	4131	3.98	5962	6201	6121	3.92

The first problem from using the cube molds to calculate SAI comes from determining the compressive strength of control mixes. At 7 days, OPC-2 and OPC-3 did not meet the 8.7% of average specified permissible range, with OPC-2 having the highest value omitted and OPC-3 having the lowest value omitted. Then at 28 days, both OPC-2 and OPC-3 had three cubes within the permissible range, while OPC-1 had the specimen with the lowest strength out of the permissible range. Taking into account the fact that these mixes are straight cement mortar cubes and that the results are the control for mixtures with SCMs, the precision of SAI testing with cubes needs improvement. The cylinder specimens, on the other hand, displayed a much more precise result with the three OPC mixes. Not only did the three mixes all fall within the permissible range by themselves, but they also met the permissible range for two specimens even when the three mixes were grouped together. At seven days, the range of the eight specimens from three mixes was 5.8% of the average; and at 28 days, the range was 1.9% of the average. For mixtures that were tested with cubic specimens, 10 out of 17 mixes failed to display results within the permissible range at 7 days and 4 out of 17 mixes failed at 28 days. With cylindrical specimens, all 17 mixtures displayed results within the permissible range at both 7 and 28 days.

From the results presented in Tables A.1 and A.2, the 17 mixes were tested for statistical analysis. Precision at 7 days and 28 days was treated separately for analysis. At 7 days, 10 out of 17 mixes had a test result omitted due to precision. Comparing the same data for cylinders, none of the mixes had the same issue. Fisher's exact test of independence was used to analyze the contingency tables presented in Table A.3 and Table A.4. Table A.3 is the contingency table at 7 days, and Table A.4 is the contingency table at 28 days. Fisher's exact test of independence is employed when sample sizes are small, and calculates the significance of the deviation from a null hypothesis (e.g., P-value). The rows represent the type of the specimen (cubes and cylinders) and the columns represent whether or not the compressive strength results meet the permissible range from ASTM C109. In this case, if the resulting two-tailed P-value is smaller than 0.05, the result would indicate that the association between rows and columns is considered to be statistically significant; which in turn proves that using cylinders gives better precision than using cubes.

Table A.3: Contingency Table at 7 Days for Cubes and Cylinders

7-day	Pass	Fail	Total
Cubes	7	10	17
Cylinders	17	0	17
Total	24	10	34

Table A.4: Contingency Table at 28 Days for Cubes and Cylinders

28-day	Pass	Fail	Total
Cubes	13	4	17
Cylinders	17	0	17
Total	30	4	34

The resulting two-tailed P value from Table A.3 is 0.0003 with the method of summing small P values. The result indicates that the association between rows and columns is considered to be extremely statistically significant, which in turn means that using cylinders is in fact more accurate than using cubes. The same analysis was conducted for 28 days. The resulting two-tailed P value at 28 days is 0.1026. The association between rows and columns is considered to be not statistically significant. However, cylindrical specimens were chosen in favor of modified SAI testing for this study due to the precision of the control value and the statistical significance at 7 days. Furthermore, beyond the improved repeatability, cylindrical molds are readily available in abundance and cost a fraction of cubic molds (approximately \$0.25 per cylindrical mold, as opposed to approximately \$250 to \$650 for cubic molds) (Pigeon, 2015). Also, the test procedure itself is much less labor intensive due to the fact the cylindrical molds are cheap and disposable. Maintaining the condition of cubic molds after each testing takes time and labor, and validating conformance to permissible variations of specimen molds specified in ASTM C109 is also difficult in laboratories.

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